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INVESTMENT CASTING OF COLUMBIUM ALLOYS

REM METALS CORPORATION

PREPARED FOR
ARMY MATERIALS AND MECHANICS RESEARCH CENTER

OCTOBER 1975

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In work supported by the Army Materials and Mechanics Research Center, the state of the art of columbium alloy investment castings has been advanced. In addition to demonstration of castability of four different alloys, the cast material itself has been characterized with respect to microstructure, alloy segregation, weldability, and the effect of heat treatment. Based upon a number of alloy selection criteria, one of the alloys, C-103, was chosen for more detailed investigation including casting of solid and cored turbine vanes having a wall thickness as low as 0.040". Heat treatment parameters

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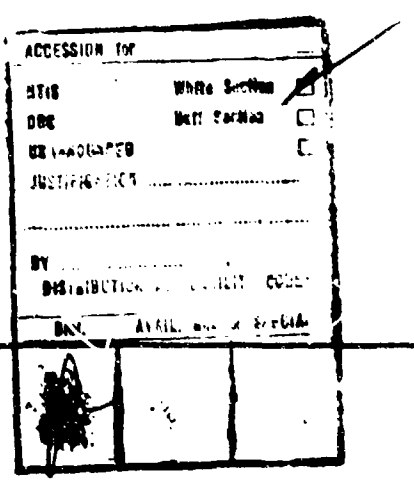
were established for optimization of C-103 mechanical properties including post-weld heat treatment. Under some casting conditions, the as-cast mechanical properties are equivalent to wrought properties. Heat treatment improves particularly ductility, and restores properties after welding. Chemistry, mechanical property, and economic considerations of material recycle are discussed. A tentative specification has been written covering C-103 castings incorporating producibility and material characterization available to date.



INVESTMENT CASTING OF COLUMBIUM ALLOYS

October, 1975

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Prepared for:

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FOREWORD

This technical report covers work performed under contract number DAAG46-75-C-0007 from August 26, 1974 through July 26, 1975. This work was performed by Rem Metals Corporation, Albany, Oregon 97321, under the technical supervision of Mr. Stanley Lopata and Dr. Robert D. French, of the Army Materials and Mechanics Research Center, Watertown, Massachusetts 02172.

This effort was part of an overall effort funded by the U.S. Army Aviation Systems Command to advance the state of the art in producibility, coatability, and practical application of columbium alloy turbine engine components.

Technical assistance was supplied by Mr. Jerry F. Koon, Metallurgical Consultant to Rem. A major portion of the metallurgical testing was subcontracted to Koon-Hall Testing Corporation, Albany, Oregon 97321.

Abstract

In work supported by the Army Materials and Mechanics Research Center, the state of the art of columbium alloy investment castings has been advanced. In addition to demonstration of castability of four different alloys, the cast material itself has been characterized with respect to microstructure, alloy segregation, weldability, and the effect of heat treatment. Based upon a number of alloy selection criteria, one of the alloys, C-103, was chosen for more detailed investigation including casting of solid and cored turbine vanes having a wall thickness as low as 0.040". Heat treatment parameters were established for optimization of C-103 mechanical properties including post-weld heat treatment. Under some casting conditions, the as-cast mechanical properties are equivalent to wrought properties. Heat treatment improves particularly ductility, and restores properties after welding. Chemistry, mechanical property and economic considerations of material recycle are discussed. A tentative specification has been written covering C-103 castings incorporating producibility and material characterization available to date.

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INTRODUCTION

There is a continuing need to improve operating efficiency of turbine engines by increasing operating temperatures. Conventional materials are being used to the limit of their temperature capability. Use of columbium alloys as turbine vane materials would allow increases in operating temperatures significantly above 2000°F.

There are two fundamental problems encountered in the use of columbium for this type of application. One of these is the necessity for providing a sufficiently effective coating to prevent oxidation of columbium at the operating temperature. Advances in coating technology are being made as a result of several programs including some especially successful work supported by AMMRC (DAAG46-74-C-0089 and DAAG46-74-C-0097). The coating problem was not addressed to any significant extent during this casting development program.

A second deterrent to the use of columbium hardware is the difficulty in obtaining the desired shape at a reasonable cost. Use of the precision casting technique for casting blades and vanes had a revolutionary effect on the economics of turbine engine manufacture in more traditional materials. Application of the precision casting process to production of columbium airfoils is anticipated to offer similar economic advantages when compared to other fabrication methods.

While the first columbium investment castings were produced six years ago, lack of applications for which the hardware could be successfuly coated and used had a slowing influence on development of a production capability for cast columbium parts. A previous effort involving AMMRC and Avco-Lycoming was instrumental in establishing the basic information on castability of columbium alloys, effect of foundry practice, and material properties. Additional effort is still needed before the characteristics of investment cast columbium alloy parts are fully defined. Full advantage of the cost savings to be achieved will require primarily an increase in the volume of production. Significant increases in volume can only come about through an expanded knowledge of the properties and behavior of the cast components, and through coatings which will allow reliable application of the parts in production engines. With acceptance of the first columbium alloy component for the F-100 engine, the industry is one major step closer to practical applications of cast columbium alloy components.

The program reported herein was performed as part of the overall effort to establish a more complete characterization of columbium investment castings, and to further expand the state of the art in this important area of technology.

The program was carried out in essentially two phases. Firstly, test specimen castings were produced for comparison of properties, structure, etc. of four columbium alloys which are potentially useful

in the investment cast form. Following this limited evaluation and comparison of the alloys, one alloy was selected for further investigation including casting of a typical vane configuration. Thorough examination of the processing parameters and the resulting cast product is summarized in this report.

Investment casting, or "lost wax" casting has been known and used since the time when all interactions of matter were explained in terms of the four "elements": earth, air, fire, and water. Continual expansion of the variety of materials and the conditions for their use place an ever increasing burden on the casting supplier to know how to handle such exotic materials as columbium and on the user to be able to intelligently select the best material for his needs. The work reported herein is considered a significant advancement in both areas of foundry capability and product characterization.

SECTION II

PROCESS OUTLINE

When reduced to its essentials the process for investment casting of columbium alloys is very similar to that for other metal systems. The high melting temperatures of these alloys, extreme reactivity toward other materials, and sensitivity to contamination by impurities all place special restraints on the specific processing steps and techniques which may be employed. The following discussion includes a general picture of the investment casting process and how the specific characteristics of columbium alloys affect the process requirements.

TOOLING DESIGN AND FABRICATION

Once a component configuration is selected, the first step in the investment casting process is fabrication of tooling. Wax pattern injection tooling consists of a split die with a machined cavity having the same shape as the final casting desired.

The material commonly used for wax injection tooling is aluminum. The material cost as well as the machining cost of the tool is lower than when steel is used. The main advantage to be gained by use of steel tooling is a longer operational life, but the useful life of an aluminum tool usually far exceeds the requirements for most aerospace castings.

The cavity in the injection tool is slightly larger dimensionally than the final casting to allow for shrinkage of the wax pattern, shell, and of the casting during cooling. The shrinkage factor required for columbium is higher than typical for other materials, at least partially due to the greater spread between the solidification temperature and room temperature. A more detailed discussion of shrinkage actually experienced on program castings will be given in Section X.

PATTERN MOLDING AND ASSEMBLY

There are a number of commercially available pattern materials which have been used successfully for preparation of columbium molds. A pattern wax may actually consist of six or more components designed to give the best combination of melting range, flow, shrinkage, surface finish, and dimensional stability. Molded patterns used in this program were prepared using McCaughin 2P31 pattern wax. Gates, runners, etc. are prepared from lower cost waxes since dimensional stability is not as important for these parts of the mold.

Following molding of the patterns, the next step is the preparation of a pattern assembly. If the patterns are small, as is usually the case, a multiplicity of them are attached to a gating and

risering system. If the pattern is particularly large, only one pattern will be attached to the gating and risering system. The gating and risering system may consist of only a simple downsprue, or may consist of a complicated system of risers, runners, ingates, and downsprues. Assembly of the pattern is performed using wax welding techniques that are common in the investment casting industry.

Following completion of the assembly operation, the assembly is inspected to assure that it was prepared according to the technique card for the specific part number, that all joints are adequately sealed and blended, and that there are no defects in the assembly that will result in any requirement to repair the castings produced.

MOLD PREPARATION AND CURING

Up to this point, all processing operations are very similar for columbium and other types of castings. The investment, or building up of the mold itself, is markedly different with respect to the materials used. Most traditional metals can be poured quite satisfactorily into molds prepared from low cost, readily available ceramics such as silica or aluminosilicates. In addition to reactivity problems, these materials are unsuitable for columbium because their melting point is below the melting point of the metal to be poured.

The techniques for building up the shell are fairly similar to other systems, the primary difference being the materials. Columbium molds are prepared according to a proprietary formulation as described in U.S. Patent number 3,422,880 and related issued and pending patents. The first dipcoat applied to the wax pattern assembly contains tungsten and a tungsten forming binder system, which provides a non-reactive lining in the finished shell. Typically, a total of eight dipcoats will be applied to the wax pattern assembly, with appropriate drying time between dipcoats and after the final dipcoat.

Removal of the wax pattern takes place in a "degreaser" using trichlorethylene as the heat transfer medium as well as solvent.

The hollow mold is then baked out at a low temperature (ca. 500^{o}F) followed by firing in a hydrogen atmosphere at a temperature in excess of 2000^{o}F. This firing cycle performs the dual purpose of removing volatile residual pattern material from the mold, and chemically curing the solid binder systems to a maximum strength.

Between the time firing is complete and it is to be cast, the mold is stored at 150-200^{o}F to minimize any pickup of moisture which could cause contamination of the mold or outgassing when the mold is preheated or cast.

MELTING AND CASTING

Other than the mold system, the most critical ingredient in production of a columbium investment casting is the melting and pouring cycle.

Prior to initiating the melt, the mold is preheated in vacuum to the desired temperature for pouring. The melting method used is the consumable electrode arc technique. The metal to be poured is purchased in the form of electrode stock, which is loaded into the furnace and attached electrically to the negative side of the DC power supply system capable of delivering approximately 500,000 watts at a current of 15,000 amperes. The positive side of the circuit is made through a water cooled copper crucible. An arc is struck between the electrode and the crucible and the power so generated melts the metal very quickly. When sufficient metal is melted, the mold is removed from the preheat chamber, the electrode is removed from the crucible, and the pour is made. The complete melting and casting process takes place in vacuum, which is maintained until the casting is cooled sufficiently to remove it from the furnace. The vacuum can be replaced by an inert gas backfill to expedite cooling.

CLEANING AND FINISHING

After the mold is removed from the casting furnace, the next step is removal of the mold and the gates and risers to expose the casting. Removal of the shell is begun using a pneumatic hammer where it can be used without damaging the part, and by hand using a hammer and chisel where required. The knockout operation is in no way unique to columbium castings, the same technique is being used on investment castings of other metals by Rem and other foundries.

Following shell removal, the castings are removed from the gates, risers, etc. using a bandsaw or abrasive wheel. Before leaving the cleaning area, one additional sequence is required which removes residual ceramic material as well as the tungsten which tends to remain on some of the casting surfaces during normal knockout operations. First, the casting is shot blasted using steel shot to remove loosely adherent material. By completion of this step, essentially all of the ceramic and most of the tungsten mold liner has been removed. The casting is then immersed in a fused salt bath for final removal of the tungsten. The fused salt used is a proprietary composition purchased from Kolene Corporation under the trade name "DGS". It is basically sodium hydroxide with additions to produce an oxidizing condition in the bath. The oxidizing salt bath reacts with the tungsten to form sodium tungstate, while no appreciable reaction with the columbium metal itself occurs (operating temperature ca. 700°F). Following caustic cleaning, the casting is given a light pickle in a dilute nitric/hydrofluoric acid solution to neutralize residual caustic and facilitate complete rinsing of the part.

Final gate removal is accomplished by machining wherever possible, leaving the maximum thickness of the remaining gate as specified by the particular component drawing. Where machining is either impossible or impractical, gate removal is completed by grinding and blending with abrasive belts or carbide grinding tools.

INSPECTION

Radiographic, penetrant, visual, and dimensional inspection are carried out on the final parts in a manner very similar to other types of castings. Detailed inspection results on the castings produced will be discussed subsequently in this report.

SECTION III

CASTING OF ALLOY EVALUATION TEST SPECIMENS

Two fundamental kings of castings were poured during this program. First, test specimen blanks were poured for use in comparison of four potential casting alloys with respect to castability and properties. Secondly, castings were produced of a mockup vane configuration having characteristics similar to those expected in a practical turbine vane component. Test specimen pours described in this section were carried out while tooling was being fabricated for the more complex turbine vane configuration.

Since all specimens would be machined prior to testing, test specimen shapes were chosen which utilized readily available tooling from which the final specimens could be machined.

MOLD PREPARATION

Wax pattern assemblies were fabricated containing both round and flat test specimen blanks for each alloy. Section thickness of the flat blanks was 0.090". Two diameters of round bar were cast. Most round specimens were nominal one-half inch diameter which was adequate for tensile specimens. Two five-eighths inch diameter blanks were also included for each alloy to enable machining of oxidation-erosion specimens from each alloy if desired. One of the wax pattern assemblies for the test specimens is shown in Figure 1.

Using these pattern assemblies, molds were prepared using investment, dewax, and firing techniques described in the Process Outline Section. Figure 2 shows a completed mold ready for loading into the casting furnace.

MELT STOCK PREPARATION

As described in the process outline, a consumable electrode must be prepared for each alloy composition to be cast. The melt stock has as its primary requirement that the chemistry be as specified, and secondly, that the material be in a form that it can be successfully melted.

A photograph of the melt stock for each alloy is shown in Figure 3. "ML" numbers throughout this report each refer to a "metal lot", or material from a single master heat.

The variability in form is due to the form of material which was available at the time of purchase. For example, the SU-31 was produced by melting a standard size ingot and then quartering it to give a small enough cross section to melt in the crucible which was used. The C-129Y was available as rejected bar stock material and was large enough that sectioning was also required. The Cb-752 and C-103 were rejected bar stock of a small enough size that further

cutting was not necessary. In cases of using previously rejected material, the cause for rejection must be reviewed to assure that the castings which result will be unaffected. The cause for rejection of previously rejected bar stock used for remelt in this program was internal ultrasonic indications. The main factor affecting the castings is the chemical composition of the melt stock, which is not affected in any way by closed internal discontinuities in the melt stock.

The use of melt stock of varying geometries would seem to introduce an additional variable into the alloy comparison. However, previous experience including use of many varying types of electrodes of the same alloy have shown electrode configuration to be a second or third order variable and would not detrimentally affect the comparison of different alloys.

Figure 4 shows the three types of electrode stock after assembly of the electrode. The electrode pieces are welded together as necessary and then welded to an attachment stub by TIG welding in a dry box which has been evacuated and then backfilled with argon. The use of a standard stub end facilitates attachment to the "stinger" or negative electrical conductor of the furnace.

MELTING AND CASTING

For all alloys except C-129Y, the melting and casting operation was relatively normal. For C-129Y, two abortive pour attempts occurred before the successful pour. Information on the metal charge, final weights, and melting parameters is tabulated in Table I.

All melts except the final C-129Y melt were performed with a crucible having a brimfull capacity on the order of fifty pounds of columbium. The crucible used for the successful C-129Y melt has an approximate capacity of 100 pounds of columbium.

The vacuum levels shown are typical of what is achievable on a production basis and are adequate for melting and casting. The relatively high pressure rise occurring with Cb-752 is attributed to differences in furnace conditions or electrode cleaning rather than to basic alloy composition.

Power input currents are shown as the maximum steady current during the melt. The melt is typically initiated with a current of 3,000 amps at approximately thirty volts. Current and, to a lesser extent, voltage are increased as quickly as possible as the molten pool is established and increased in size. The melt is continued until the desired quantity of metal is melted, with the variation in maximum input power depending on the melt weight and electrode configuration.

In the case of C-129Y, melting problems were encountered which appeared to be specific to this alloy. The initial melt was started in the same manner as melts of the other alloys. However, instead of establishing a normal melt, little melting occurred. In this situation, the arc became localized on one area of the crucible resulting in melting of the copper and perforating it such that the cooling water was allowed to enter the crucible.

Chemical reaction of the cooling water with the molten or heated columbium resulted in contamination of the material with oxygen and hydrogen. The material in the crucible was discarded, and the electrode cleaned to remove the contaminated end prior to attempting another melt.

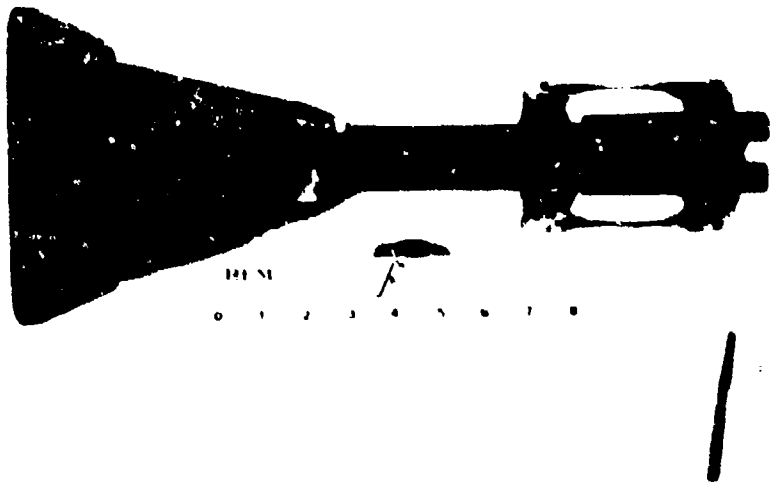
After repair of the crucible liner, a second melt attempt was initiated, using a greater quantity of starting material in the crucible. Even after a longer melt time than required for the complete melt of the other alloys, a full pool covering the bottom of the crucible had not been formed. In this case, no material loss or contamination occurred and the same melt stock and starting chips were used for the next trial.

Based upon the above results and consultation with personnel at the material source relative to experience in arc melting C-129Y ingot, the conditions were set for what turned out to be a successful melt. Rather than using the normal 3,000 ampere starting current, a much higher starting current (8,000 amperes) was selected. This necessitated the use of the larger crucible to increase the clearance between the electrode and crucible and preclude the possibility of arcing to the crucible sidewall with the higher start-up power. Once a pool was established, melting proceeded very quickly and without additional difficulty.

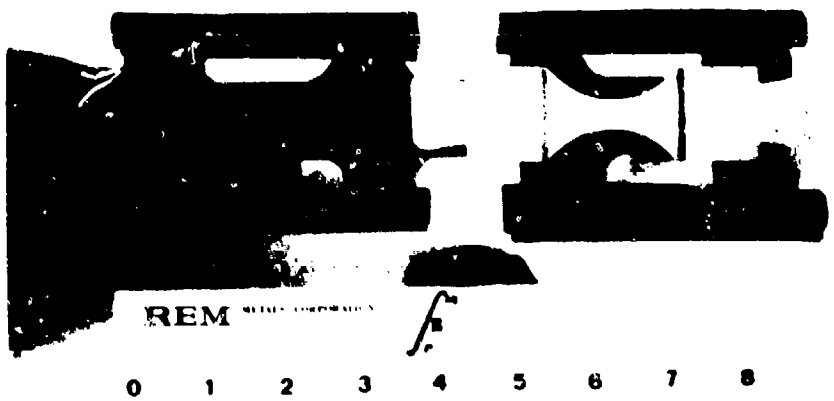
The SU-31 mold after pouring, shown in Figure 5, is typical of the appearance of the molds after casting. The castings of each alloy are shown in Figure 6 after most of the mold has been removed by mechanical means. Figure 7 shows the appearance of the individual test specimen blanks after being cut off from the downsprue.

CASTING EVALUATION

The castings were thoroughly examined for chemistry, NDT quality, and mechanical properties. A detailed discussion of these specific evaluations are presented in other sections of this report.



Front View Showing Pouring Cup



Side View

Figure 1. Wax Pattern Assembly Configuration for Alloy Evaluation Castings

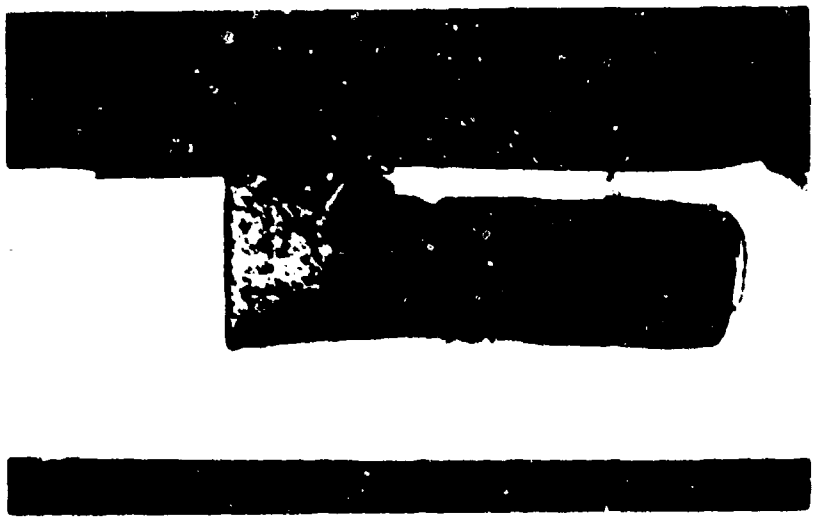
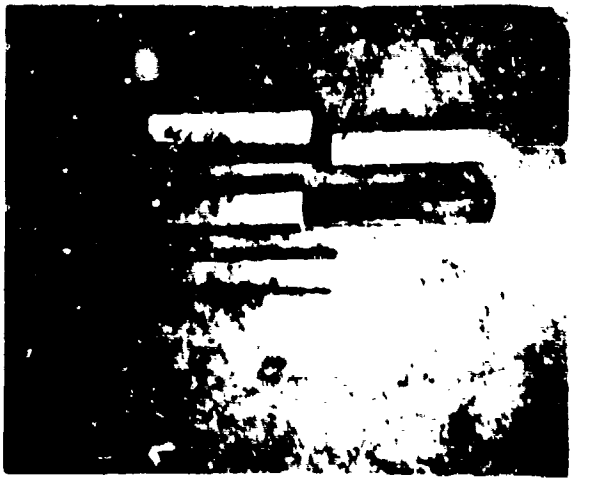


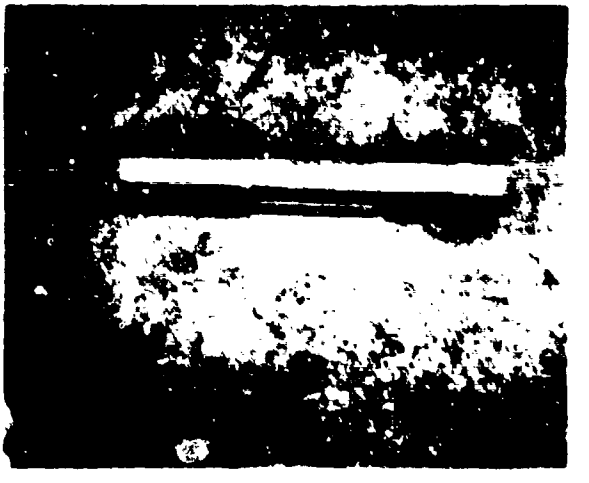
Figure 2. Completed Test Specimen Mold, Ready for Casting

C-129Y, 47.4 Pounds
ML 662



SU-31, 50.5 Pounds
ML 179

C-103, 40.0 Pounds
ML 648 (Long Piece)
ML 649 (Short Piece)



Cb-752, 40.2 Pounds
ML 650

Figure 3. Melt Stock for Alloy Comparison Pours

Cb-752 Electrode, 43.0 Pounds

SU-31 Electrode, 48.5 Pounds

C-129Y Electrode, 43.0 Pounds

Figure 4. Electrodes Assembled by TIG-Welding Melt Stock to A Stub

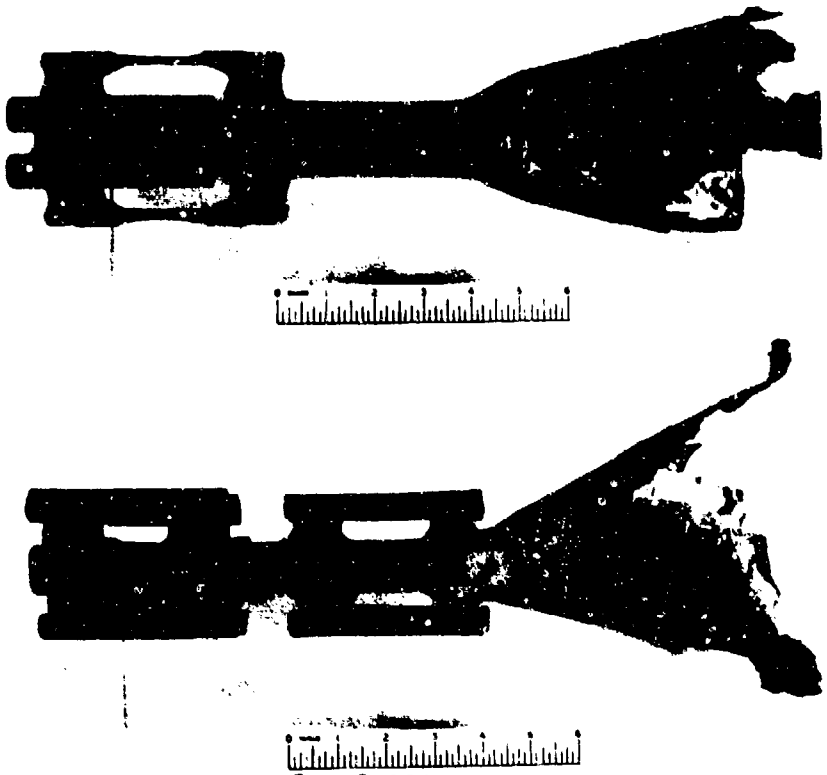
TABLE I

MELTING PARAMETERS FOR ALLOY EVALUATION POURS

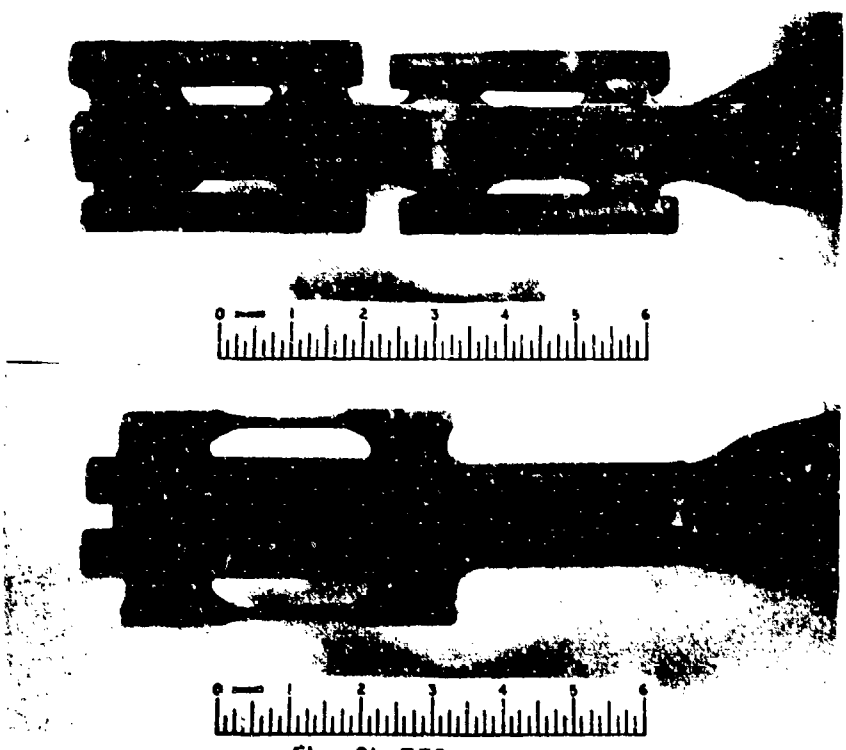
	C-103	Cb-752	SU-31	C-129Y		
				Attempt #1 (1)	Attempt #2 (2)	Successful Pour
Crucible Size	6" dia.	6" dia.	6" dia.	6" dia.	6" dia.	8" dia.
Crucible Charge Wt.	20.5 lbs.	2.5 lbs.	2.25 lbs.	3 lbs.	4 lbs.	9 lbs.
Vacuum at Start of Melt	7μ	10μ	3μ	2μ	7μ	4μ
Vacuum Rise During Melt	8μ	40μ	5μ	---	---	10μ
Furnace Hot Leak Rate	13μ/min.	20μ/min.	19μ/min.	20μ/min.	---	9μ/min.
Final Skull Wt.	27 lbs.	10.5 lbs.	12.25 lbs.	4 lbs.	8 lbs.	12 lbs.
Wt. Melted from Electrode	22.5 lbs.	32.25 lbs.	36 lbs.	1 lb.	4 lbs.	27.25 lbs.
Weight Poured	16 lbs.	24.25 lbs.	26.0 lbs.	None	None	24.25 lbs.
Power Input (Maximum amps)	5000A	8000A	6000A	4000A	5000A	11000A
Voltage (maximum volts)	32	35	40	34	35	38
Power (Kilowatts)	160	280	240	136	175	418
Melt Time	5 min.	7 min. 36 sec.	8 min. 36 sec.	1 min.	8 min. 48 sec.	2 min. 24 sec.
Time to Pour	2.8 sec.	2.8 sec.	2.55 sec	---	---	3.1 sec.
Mold Temperature	1000°F	960°F	1010°F	---	---	1000°F

(1) Aborted due to perforation of the crucible.
(2) Aborted due to failure to establish a molten pool.

Figure 5. SU-31 Mold After Casting and Before Knockout

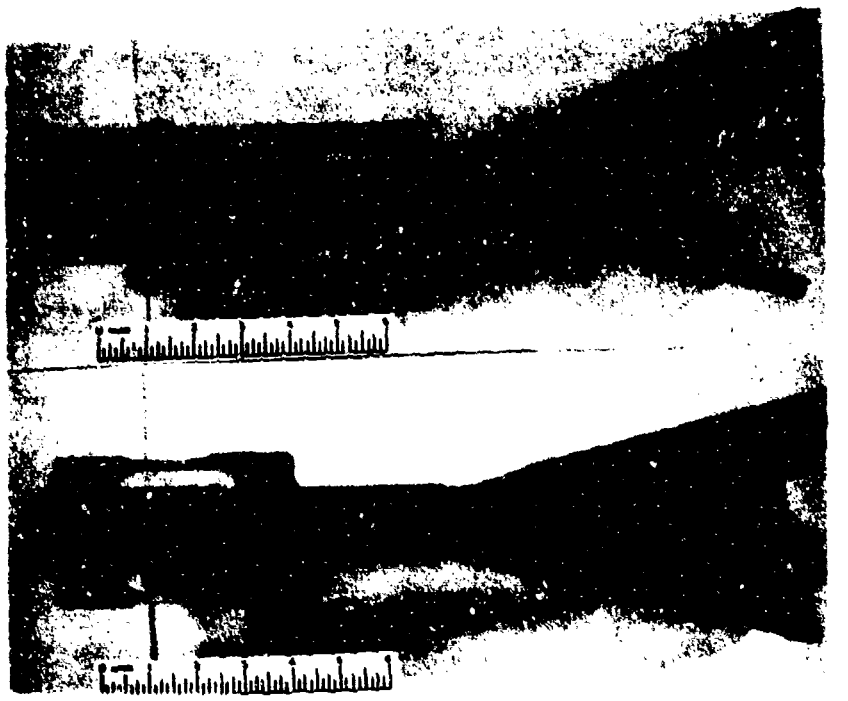


6a. C-103

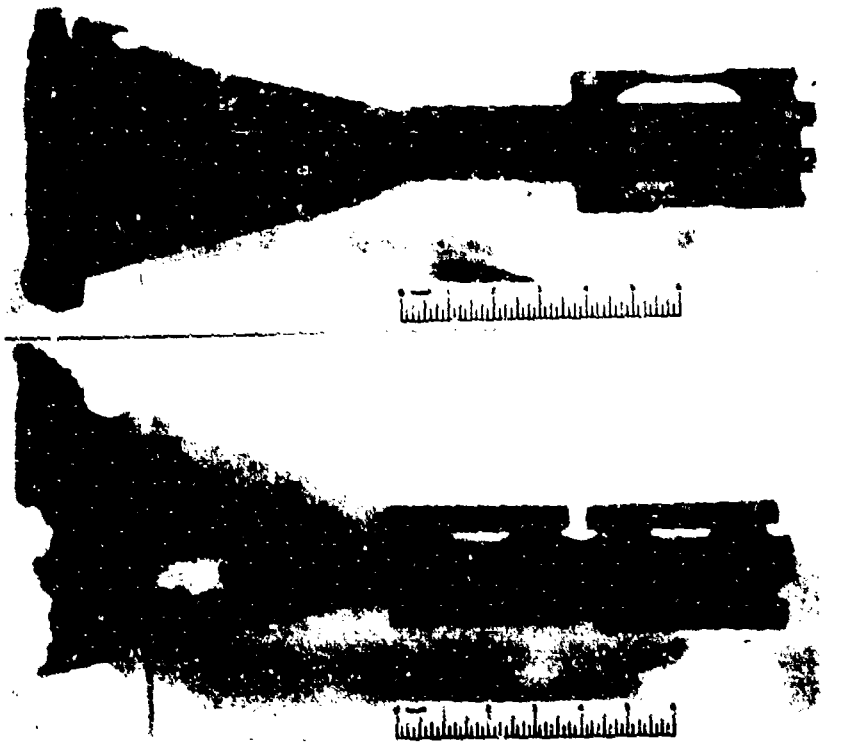


6b. Cb-752

Figure 6. Test Specimen Castings After Mold Removal



6c. SU-31



6d. C-129Y

Figure 6 (Cont'd.) Test Specimen Castings After Mold Removal

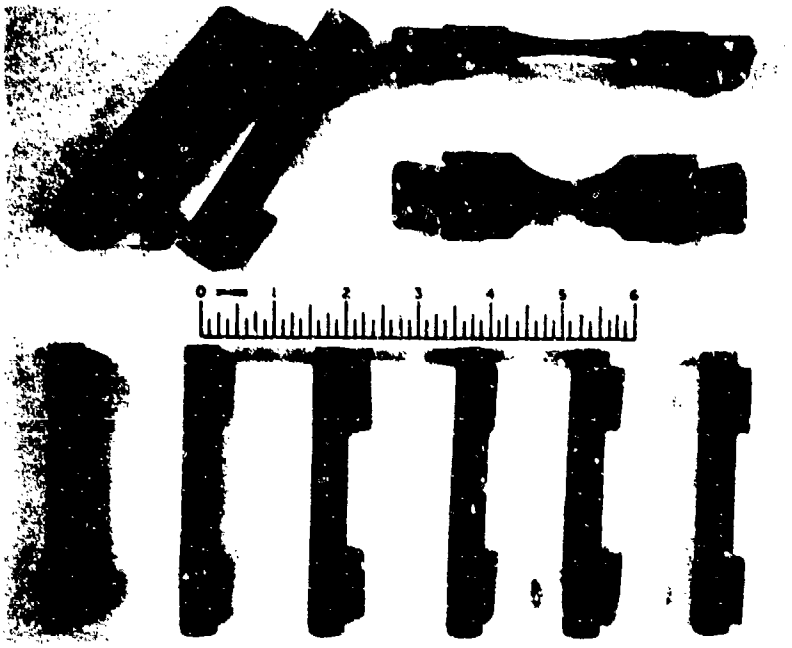


Figure 7. C-103 Castings After Cut Off

SECTION IV

CHEMICAL ANALYSIS

Characterization of the chemical composition of the materials used is of the highest order of importance in understanding or being able to reproduce the results of an effort such as this.

Table II shows the composition of a number of columbium alloys which have been or are currently available on a somewhat commercial basis. There are a great variety of other alloy compositons which have been proposed or tested which have certain advantages in the areas of properties, workability, or oxidation resistance. However, since alloy development is outside the scope of this program, the only ones seriously considered were those for which a reasonable backlog of behavior information was available.

In selecting a columbium alloy composition for investment casting purposes, the approach to date has been to choose from wrought alloys rather than attempting to change composition to maximize castability or casting properties. As more is learned about the relationship of chemistry and properties, there will undoubtedly be adjustments to existing alloy compositions as well as new alloy systems.

Program ingot/electrode and castings were analyzed by the methods shown below:

ALLOYING ELEMENTS (Greater than 1000 ppm)

Atomic absorption spectrophotometry using Perkin-Elmer models 303 and 306.

IRON

Emission spectrographic analysis using a Baird-Atomic 3.0 meter air path instrument.

CARBON

Oxygen combustion analysis using a LECO model CS-44 with infrared readout.

HYDROGEN

Vacuum fusion using LECO model RH-1.

OXYGEN

Impulse inert gas fusion using LECO model TC-30 with chromatographic readout.

NITROGEN

Inert gas fusion by LECO model TC-30 or MicroKjeldahl distillation titration.

In order to facilitate ready reference to the data, all chemical analysis results generated during the program are collected in Appendix 1. All results shown as "ingot" refer to analyses performed prior to any conversion of the heat from ingot size to a useable size for casting. Those samples identified as "electrode" were sampled from the electrode prior to melting, while those shown as "casting" were cut from a test specimen or gate of the casting itself.

While melt stock had been procured from more than one supplier, all results shown in Appendix 1 were actually performed by a single laboratory.

Upon review of the results reported in Appendix 1, it is found that the agreement between the various analyses and in comparison with the specification limits is quite good. In the case of the interstitials, it is seen that there is a general tendency for an increase in composition level to occur both from ingot to electrode and from electrode to casting. This is a result of some degree of contamination which will occur during any elevated temperature working or handling of the ingot or electrode or casting. This increase in interstitial level does not represent a problem either in the original production of castings or in recycle of gates and risers as long as the interstitial impurities can be reliably held within acceptable limits.

Other than the minor increase in interstitials noted above, most of the variations noted in chemical analysis results are considered to be within normal analytical variation or due to sampling variation related to inhomogeneity of the original ingot.

Based upon these results, it can be concluded that the chemistry of a casting is reasonably well predictable from the ingot chemistry. This degree of consistency is an important point to have established in this or any alloy system.

TABLE II

Nominal Composition of Columbium Alloys

Alloy Designation	Cb*	W	Hf	Ti	Ta	Zr	V	Mo	C	Y
Cb-1Zr	99.0					1				
B-33	95.0						5			
D-14	95.0					5				
AS-55	94.0	5				1				
B-66	89.0					1	5	5		
D-43	88.9	10				1			0.1	
C-103	88.3		10	1		0.7				
Cb-752	87.5	10				2.5				
D-36	85.0			10		5				
SCB 291	80.0	10	10							
D-31	79			10				10	0.1	
C-129Y	79.4	10	10		0.5					0.1
SU-31	79.4	17	3.5						0.1	
15W-5Mo-1Zr	79.0	15				1		5		
XB-88	69.93	28	2						.07	
FS-82	67.0				32.0	1				
FS-85	61.0	10			28.0	1				
Cb-132M	58.4	15			20.0	1.5		5	.1	
WC 3015	54.9	15	28			2			0.1	

*The nominal columbium content is obtained by substracting the sum of the other elements from 100 (without considering the impurities such as O, N, H).

SECTION V

METALLOGRAPHIC STUDY OF HEAT TREAT RESPONSE

Prior to this program, essentially all investment cast columbium alloy test data was based on material in the as-cast condition. Characterization of the microstructural heat treatment response for the cast material has been performed for the four program alloys and is reported in this section.

Specimens for the heat treat trials for each alloy were one-half inch long sections cut from a single one-half inch diameter by four-inch long cast round bar. The specimens were taken from the first mold poured of each alloy during this program. One section of each specimen was retained with no heat treatment for simultaneous examination of hardness and microstructure.

Selection of specific heat treatment conditions was obviously based on fairly limited background information, relying heavily on data available for some of the alloys in the wrought form. The heat treat cycles, including time and temperature, which were selected for evaluation are shown in Table III. These conditions were believed to be best for determination of response characteristics and comparison of the four alloys. The solution treatments both with and without subsequent age-anneal cycles represent trials to establish the feasibility of removing all precipitates from the grain boundaries with subsequent redistribution to the grain matrix. If successful, this would be expected to result in improved ductility and stress rupture capability for any given strength level.

Heat treatment of the test specimens was carried out by Teledyne Wah Chang Albany at a nominal pressure of 0.10 microns. Argon was used for rapid cooling where gas cool is specified. See Table IV and Figure 8. The most important cooling rate comparison is that of the furnace cool versus gas cool from the 2900°F solution temperature. As intended, the gas-cooled specimens were much more effectively "quenched" then those that were furnace cooled.

The apparent anomaly in comparison of the furnace cooling rates, (i.e. cooling from 2550°F was more rapid than from either 2200°F or 2900°F), is due to differences in total mass of the load in the three runs.

After heat treatment, Rockwell B hardness was determined on each sample, with triplicate results reported in Table V.

Each specimen was mounted, prepared, and examined microscopically. Final etching of the samples was done using the following mixture at room temperature: three parts lactic acid, three parts hydrogen peroxide, two parts hydrofluoric acid, two parts nitric acid. By examination at different degrees of magnification, it was found that 600X would show structural effects most representatively. Photomicrographs are shown in Figures 9 through 12.

The metallographic samples were examined visually in addition to photomicrographs. Specific comments on each alloy and condition are shown in Table VI. Most of these observations are visible in the photomicrographs and are discussed below.

SU-31

It is known, that this alloy responds well to the temperatures used when in the wrought form. However, the response of cast SU-31 in this test was minimal. The hardness of the samples was the same, within experimental error, regardless of the heat treat condition.

The microstructure likewise did not show any apparent change after any of the five heat treat cycles. In the as-cast micrograph, we see what are presumed to be carbides at the grain boundaries, as well as semicontinuous areas of decoration within the grains which appear to cross grain boundaries. None of the heat treatments were adequate to break up or dissolve these particles. The precipitate gives some hint of being coarser in the samples which were heated to 2900°F, which would indicate they are stable and susceptable to growth at this temperature.

The temperature required to dissolve the precipitate is apparently higher than that for wrought material. It is believed that the nearly continuous precipitate at the grain boundaries is responsible for the brittle behavior of this alloy. It seems entirely feasible that a solution temperature perhaps not too far above 2900°F would be adequate to dissolve the precipitate, which could then be reprecipitated in a less continuous manner, thus imparting some additional ductility as well as strength to this alloy.

Cb-752

This alloy showed some definite changes in microstructure with heat treatment. In solution treatment at 2900°F, some of the precipitate appears to have dissolved, with no discernible difference between the two cooling rates. The 2550°F cycle appears to have the same effect.

Solutioning followed by aging at 2200°F produced a definite precipitation in the matrix similar to that seen in SU-31. Interestingly, the 2200°F cycle without previously solutioning produced the same kind of precipitate except to a lesser extent.

The hardness of Cb-752 showed some variation between samples, but the relationship between hardness and microstructure was not clear. Overall, the presence of the precipitate appears to reduce the hardness of the alloy.

C-103

This alloy responded to heat treatment somewhat differently from Cb-752. Solutionizing with a rapid cool came very close to full dissolution of precipitates. The furnace cooled material contains more frequent decoration, appearing more like as-cast than like the quenched structure.

Similarly to Cb-752, "aging" at 2200°F with or without a prior solution treatment caused substantial precipitation, more so in the sample which was previously solution treated. Unlike the Cb-752, the structure was more dense and had a stringer like appearance. Like Cb-752, the C-103 samples having the least precipitate were the lowest in hardness.

C-129Y

This alloy composition showed the same minor trend in hardness as C-103 and Cb-752. The changes in microstructure were much less pronounced, primarily affecting particles within the grains. Overall, this alloy must be considered nearly as unresponsive as SU-31.

SUMMARY

None of the alloys was fully solutionized using any of the heat treatments. Two of the alloys, Cb-752 and C-103, appear to be very close to the solution treated condition when as-cast, while SU-31 experiences substantial precipitation on cooling. An interesting correlation of heat treat response is with impurity content is shown in Table VII. The inference which could be drawn from this table alone is that the heat treat response is related to the interstitial element content rather than any other factor such as other alloy components. The total relationship obviously involves all of these factors.

In general, the lack of significant change in hardness values would indicate that heat treatment will not have the effect on tensile strength as is observed in many other alloy systems. The primary benefit of heat treatment may be related to making the strength more consistent and improving ductility by making the material more uniform.

Further investigation of heat treatment behavior was beyond the scope of the program except for limited studies on one of the alloys which are reported in Section XI.

Microprobe examination reported in the next section was used to help understand the observed heat treat response phenomena.

TABLE III

COLUMBIUM ALLOY HEAT TREAT TRIAL CONDITIONS

Heat Treat Code Number	Type of Thermal Treatment	Temperature	Time	Cooling Rate
#0	As-Cast	---	---	---
#1	Solution and Age	2900°F + 2200°F	1 Hour 1 Hour	Gas Cool Furnace Cool
#2	Solution only/gas quench	2900°F	1 Hour	Gas Cool
#3	Solution only	2900°F	1 Hour	Furnace Cool
#4	"Anneal"	2550°F	1 Hour	Furnace Cool
#5	Stress Relief or Age	2200°F	1 Hour	Furnace Cool

TABLE IV

COOLING RATE DATA FOR Cb ALLOY HEAT TREAT TRIALS

#4 2550°F/1 Hr./FC

Temperature	Minutes
2550°F	0
2400°F	.42
2000°F	.54
1800°F	1.56
1400°F	3.78
1000°F	9.90

#3 2900°F/1 Hr./FC

Temperature	Minutes
2900°F	0
2600°F	.42
2400°F	.78
2000°F	2.04
1800°F	4.08
1400°F	6.96
1000°F	12.54

#1, #2 2900°F/1 Hr./GC

Temperature	Minutes
2900°F	0
2600°F	.66
2400°F	.96
2000°F	1.38
1800°F	1.56
1400°F	3.12
1000°F	5.22

#1, #5 2200°F/1 Hr./FC

Temperature	Minutes
2200°F	0
2000°F	.54
1800°F	1.86
1400°F	5.46
1000°F	12.24

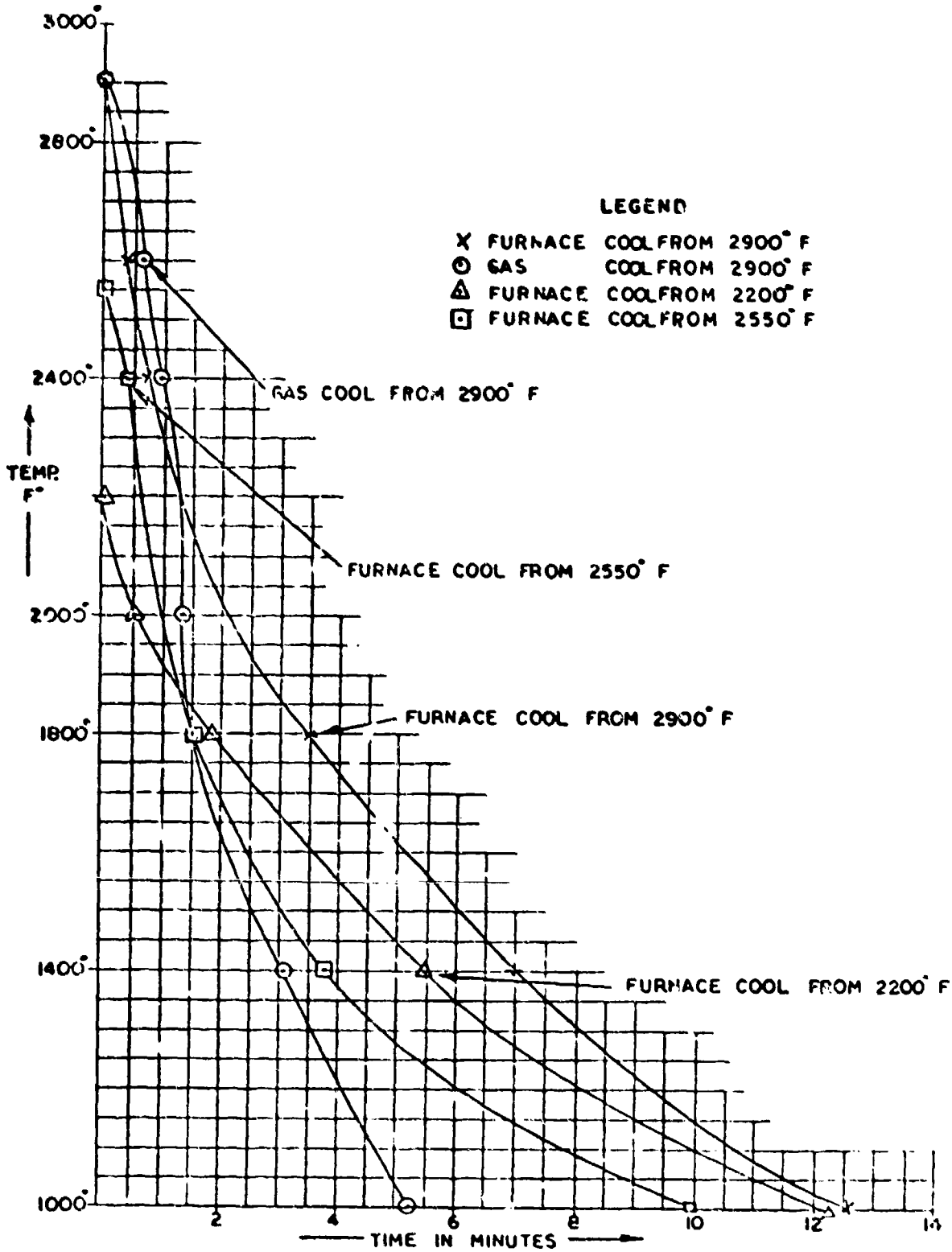


Figure 8. Actual Cooling Curves for Cb Alloy Heat Treat Trials

TABLE V

EFFECT OF HEAT TREATMENT ON HARDNESS, ROCKWELL B

Heat Treatment	C-103	C-129Y	SU-31	Cb-752
#0 As-Cast	81 80 80 80	92 92 92 92	98 99 99 99	86 88 87 87
#1 2900 + 2200	76 76 76 76	86 88 88 87	96 98 98 97	84 85 84 84
#2 2900/Gas Quench	81 81 81 81	92 92 92 92	98 98 98 98	87 88 87 87
#3 2900/Furnace Cool	83 83 84 83	91 92 93 92	98 98 98 98	88 89 89 89
#4 2500	77 78 77 77	90 90 90 90	97 97 97 97	85 85 85 85
#5 2200	76 76 77 76	88 89 90 89	98 98 97 98	85 85 84 85

600X

Figure 9a. As-Cast

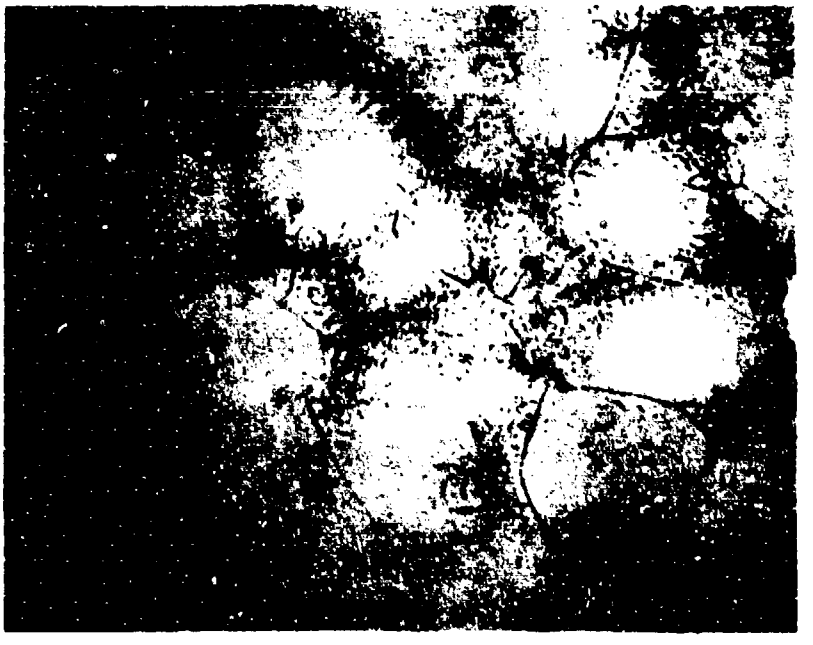
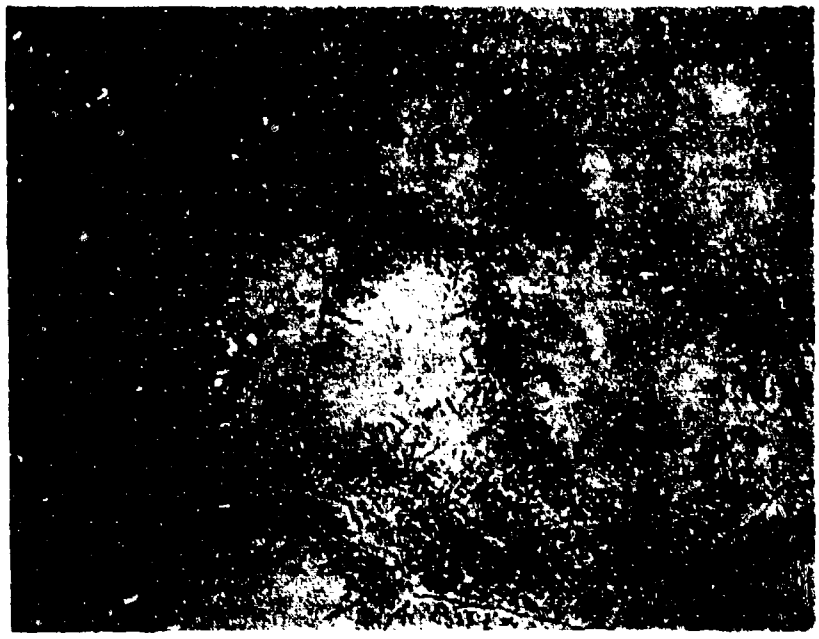


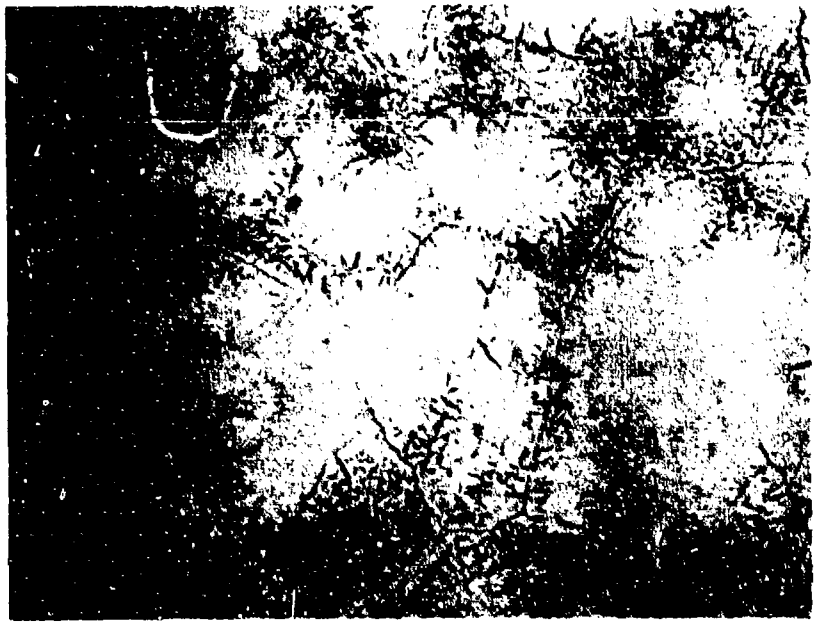
Figure 9b. 2900°F/1 Hr./GC + 2200°F/1 Hr./FC

Figure 9. SU-31 Microstructure Versus Heat Treat Condition



600X

Figure 9c. 2900°F/1 Hr./GC



600X

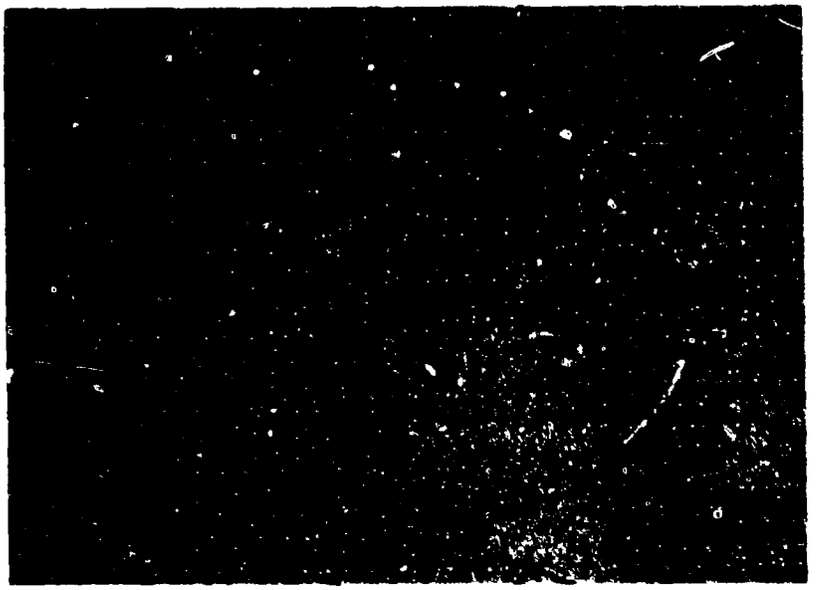
Figure 9d. 2900°F/1 Hr./FC

Figure 9. SU-31 Microstructure Versus Heat Treat Condition (Cont'd.)

Figure 9e. 2550°F/1 Hr./FC 600X

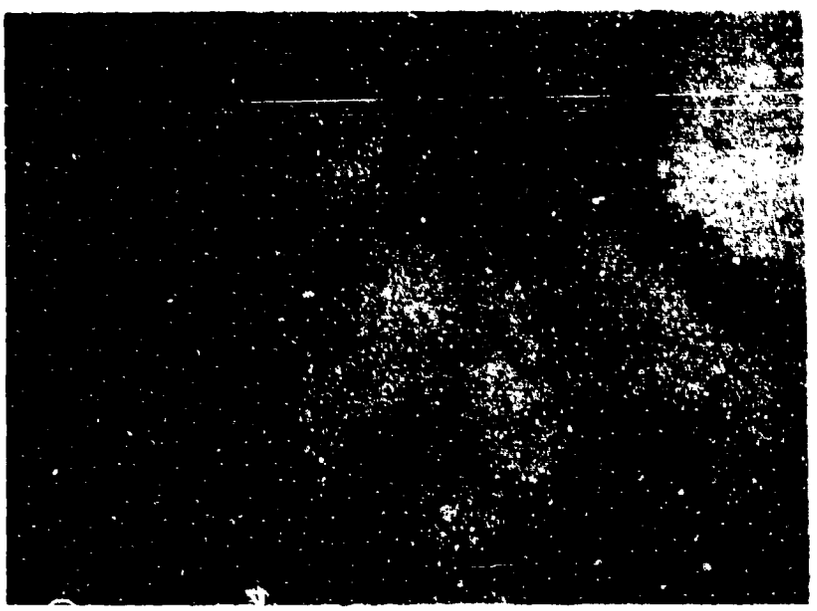
Figure 9f. 2200°F/1 Hr./FC 600X

Figure 9. SU-31 Microstructure Versus Heat Treat Condition (Cont'd.)



600X

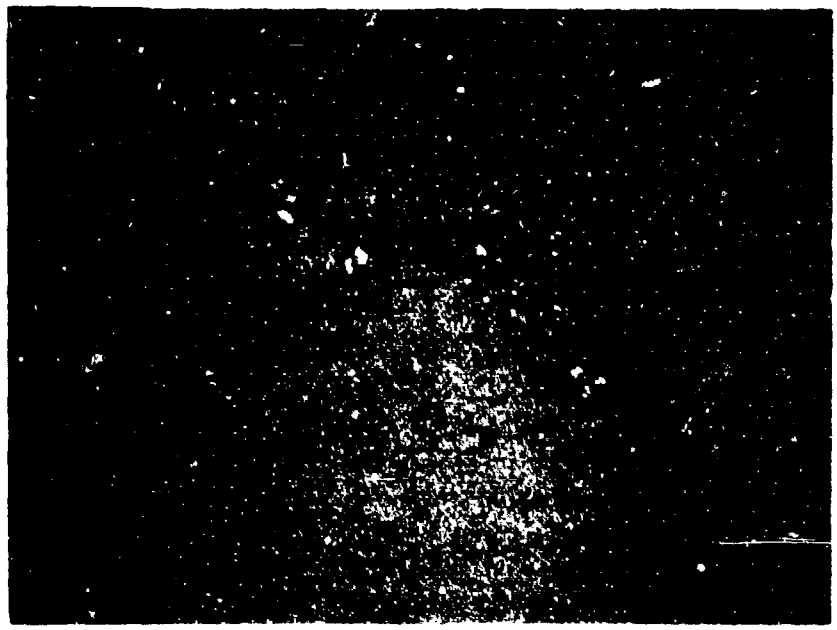
Figure 10a. As-Cast



600X

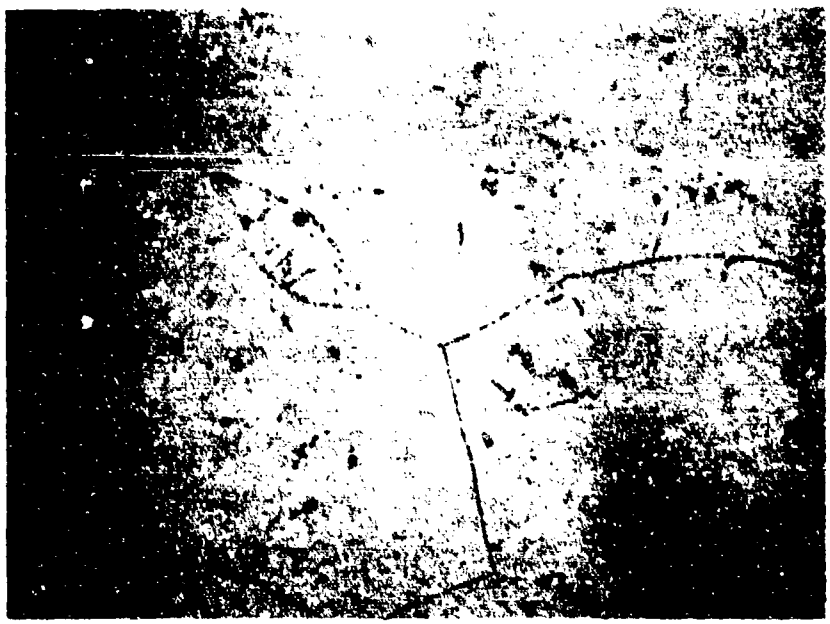
Figure 10b. 2900°F/1 Hr./GC + 2200°F/1 Hr./FC

Figure 10. Cb-752 Microstructure Versus Heat Treat Condition



600X

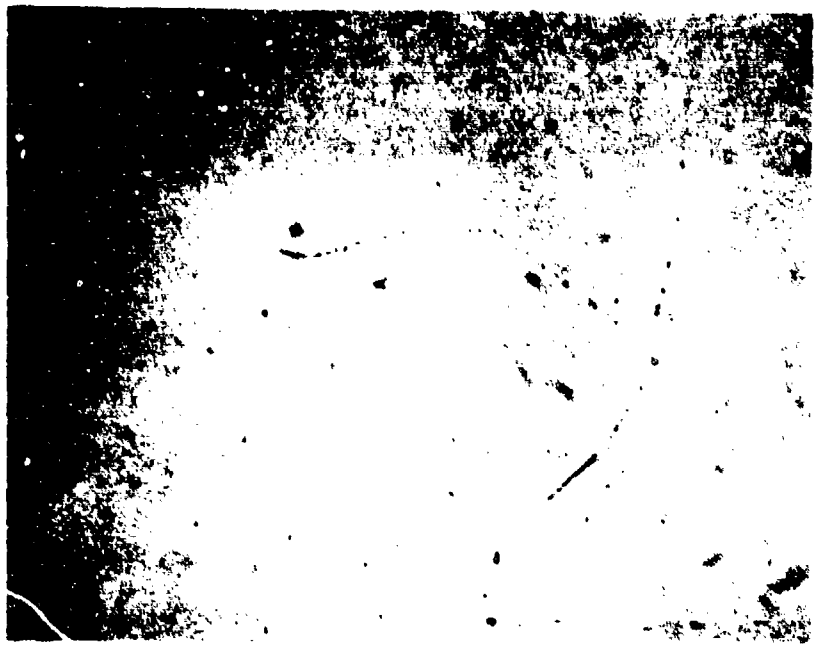
Figure 10c. 2900°F/1 Hr./GC



600X

Figure 10d. 2900°F/1 Hr./FC

Figure 10. Cb-752 Microstructure Versus Heat Treat Condition (Cont'd.)



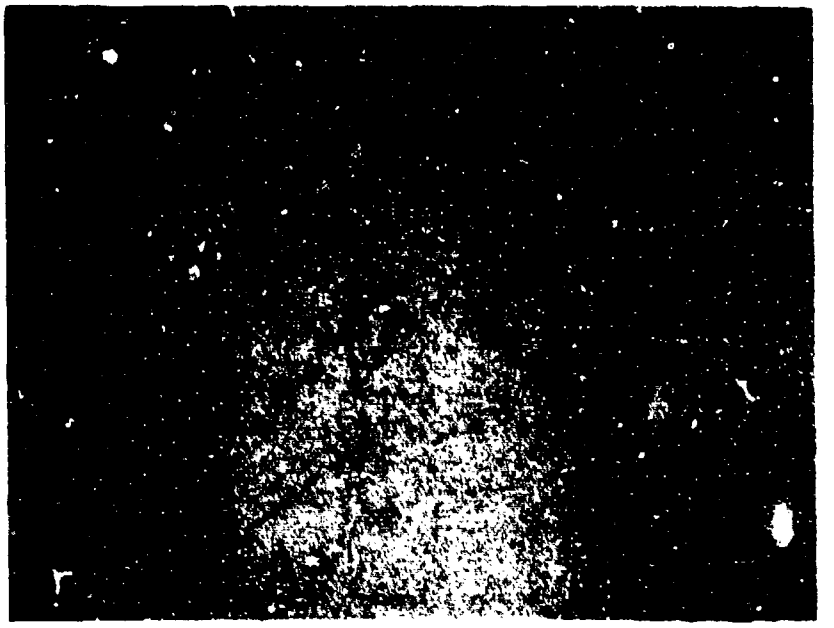
600X

Figure 10e. 2550°F/1 Hr./FC

600X

Figure 10f. 2200°F/1 Hr./FC

Figure 10. Cb-752 Microstructure Versus Heat Treat Condition (Cont'd.)



600X

Figure 11a. As-Cast

600X

Figure 11b. 2900°F/1 Hr./FC + 2200°F/1 Hr./FC

Figure 11. C-103 Microstructure Versus Heat Treat Condition

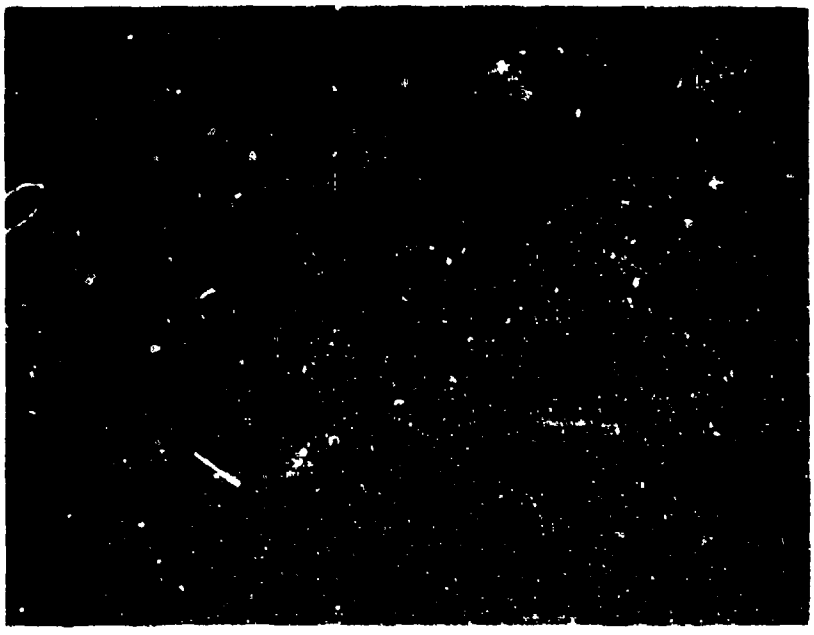
600X

Figure 11c. 2900°F/1 Hr./GC

600X

Figure 11d. 2900°F/1 Hr./FC

Figure 11. C-103 Microstructure Versus Heat Treat Condition (Cont'd.)



600X

Figure 11e. 2550°F/1 Hr./FC

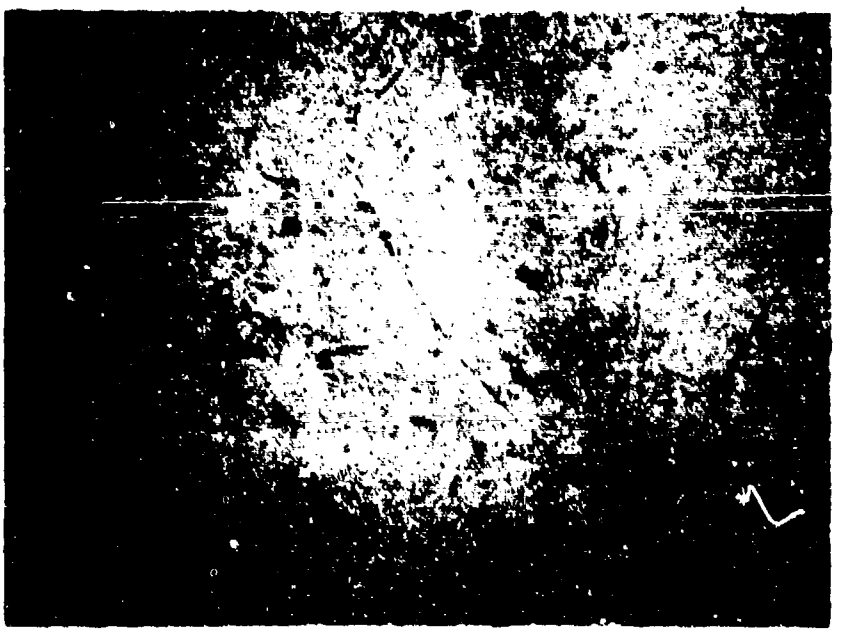
600X

Figure 11f. 2200°F/1 Hr./FC

Figure 11. C-103 Microstructure Versus Heat Treat Condition (Cont'd.)

600X

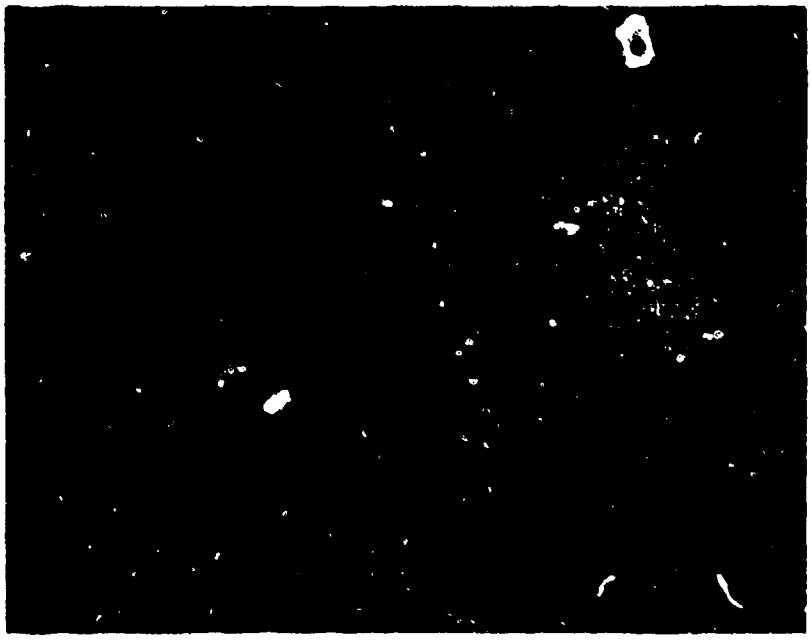
Figure 12a. As-Cast



600X

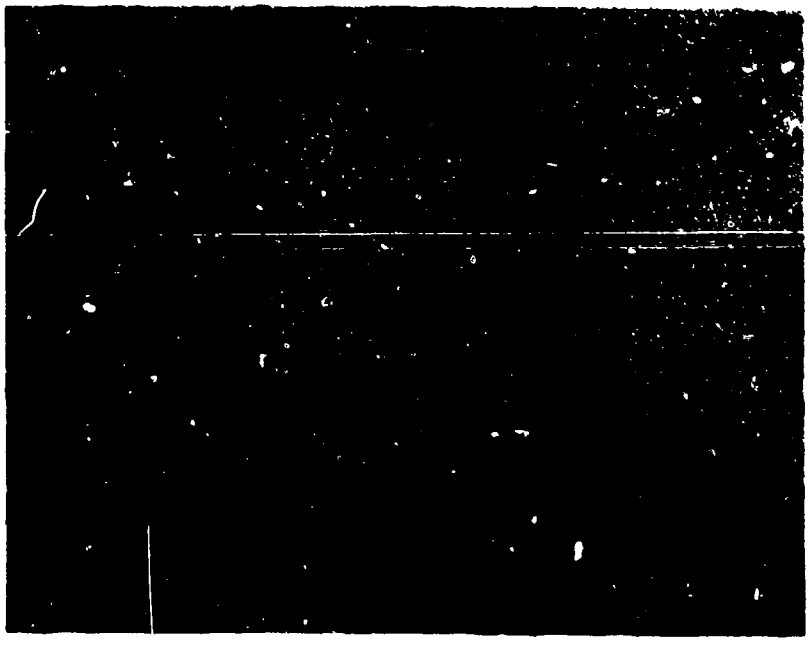
Figure 12b. 2900°F/1 Hr./FC + 2200°F/1 Hr./FC

Figure 12. C-129Y Microstructure Versus Heat Treat Condition



600X

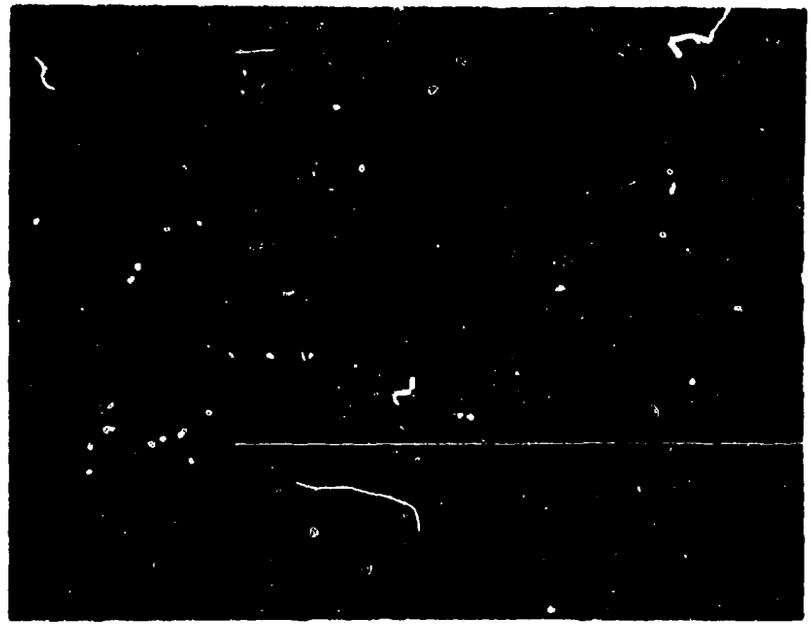
Figure 12c. 2900°F/1 Hr./GC



600X

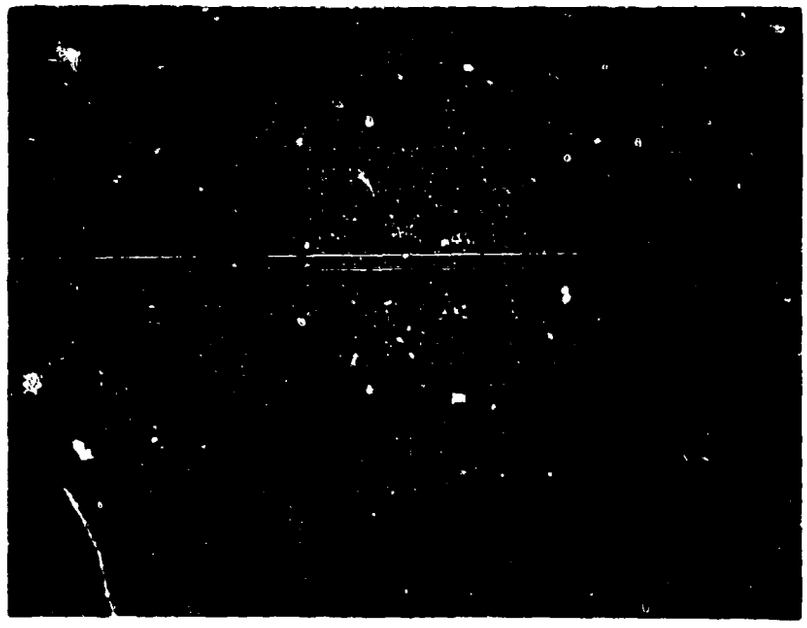
Figure 12d. 2900°F/1 Hr./FC

Figure 12. C-129Y Microstructure Versus Heat Treat Condition (Cont'd.)



600X

Figure 12e. 2550°F/1 Hr./FC



600X

Figure 12f. 2200°F/1 Hr./FC

Figure 12. C-129Y Microstructure Versus Heat Treat Condition (Cont'd.)

Heat Treat	C-103	Cb-752	SU-31	C-129Y
#0 As-Cast	Both clear and gray present both in matrix & GB's. Some gray elongated ppt at GB's.	Ppt particles located both GB and matrix. Generally fine ppt.	Very fine broken GB ppt. Significant degree of "laced" GB's. No detectible isolated matrix particles	Very small & broken ppt at GB's, very few ppt particles in matrix.
#1 2900 + 2200	Reappearance of "laced" GB's similiar to 2200/FC without prior 2900/GC. No other significant change from 2900/GC without subsequent 2200/FC age/anneal	Agglomeration of GB ppt compared to GC without/age anneal. Reappearance of "laced" GB's.	No significant change over 2900/FC without 2200/FC age/anneal. Possibly fewer clear ppt particle in matrix.	Slight agglomeration of ppt in matrix. Very few & very fine ppt at GB's particle reappearance of clusters in matrix, but of lesser extent than 2200/FC without prior 2900/GC.
#2 2900/Gas Quench	Larger, but less total number of ppt particles both GB and matrix compared to 2900/FC. Near total absence of "laced" structure. Occasional cluster of fine matrix ppt.	Less total & finer ppt than 2900/FC. Predominantly at GB's. Matrix ppt appear in clusters. Well broken at GB's.	More clear ppt particles in matrix compared to 2900/FC. GB's predominantly very fine and broken. Slightly less "laced" structure than in 2900/FC.	Fewer ppt particles at GB, but more in matrix compared to 2900/FC. More clear type particles.
#3 2550	Significant breaking & agglomeration of ppt particles. Both clear & gray types located both in matrix & at GB"s Minor amount of "laced" structure.	Significant breaking & agglomeration of ppt particles Both clear & gray types located both in matrix & at GB's Absence of "laced" GB's.	Slightly coarser GB than 2550/FC. Isolated clear ppt particles in matrix No significant change in "laced" structure.	Slight growth of ppt particles. Predominantly at GB's. Occasional larger gray angular type both in GB and in matrix.
#4 2550	Little or no matrix ppt. Less GB ppt, but approx. same particle size. Noticeable absence of "laced" structure. Ppt particles may have gone through first crystal structure change.	Less overall ppt. Noticeable absence of "laced" GB's. No signficant change in ppt particle size. Ppt particles may have gone through first crystal structure change.	Possible breaking of GB ppt in some areas. Slightly coarser GB's. "Laced" structure slightly more diffused.	Very fine & broken ppt at GB's & in matrix. Absence of clusters around GB's but more in matrix compared to 2200/FC Ppt particles may have gone through first crystal structure change.
#5 2200	Significant increase in amount of GB "laced" structure over as-cast No significant change in degree size, shape, nor location of other ppt.	Ppt at GB has agglomeratedslightly Possibly more broken.	Slightly more pronounced GB's & "laced" GB structure compared to as-cast.	No significant change from as-cast No gray type particles detected (all clear type).

TABLE VII

OVERALL HEAT TREAT RESPONSE
CORRELATION WITH INTERSTITIAL CONTENT

	Actual Cast Compostion Ppm				
	C	O	N	Total C+O+N	Heat Treat Response
SU-31	1010(1)	50	70	1130	None (Overloaded with precipitate)
Cb-752	80	210	63	353	Good
C-103	80	300	103	483	Good
C-129Y	40	110	38	188	Little Response (Very little precipitate in as cast condition)

(1) Carbon is an alloy constituent in SU-31. All other alloy/element combinations in this table are present as impurities only.

SECTION VI

MICROPROBE EXAMINATION

The electron microprobe provides a valuable tool in the elucidation of microstructural phenomena in metallic alloys. Selected samples of each of the four program alloys were subjected to microprobe examination with the intention of identification of grain boundry phases, inclusions, or alloy component segregation.

The equipment used was a Hitachi model No. XMA-5 electron microprobe instrument.

Table VIII summarizes the tests which were conducted. Most of the particles were chosen as representative of the appearance of typical particles in one of the alloys.

PARTICLE IDENTIFICATION AND ANALYSIS

The location and metallographic appearance of the several particles which were examined are shown in Figures 13 through 17. The microprobe results on the particles are summarized in Table IX and are shown with each figure along with a comparison to actual chemical analysis results which represent average chemistry of the sample. As noted at the bottom of Table IX, the weight percentage numbers calculated from microprobe intensity ratios have not been corrected for second order effects. The percentages reported are, therefore, to be considered approximate, and of the most benefit for comparison purposes.

Figure 13 shows the location of two grain boundary particles. Figures 14 and 15 show two similar particles in the matrix. Each particle is marked by arrows from the outside of the picture, the particle being located at the intersection of the two perpendicular lines drawn through the arrows. All four of the particles analyzed in C-103 (two each grain boundary and matrix) are clearly high in hafnium and oxygen, indicating basically a hafnium oxide precipitate. Carbon is also definitely high in the two non-elongated particles. Carbon in the two elongated particles was below detection limits but was presumably present to some extent above the matrix level. Interestingly, the titanium content of all four particles was below that of the matrix, while zirconium was somewhat increased.

Figure 16 shows the results of analysis on one unique particle which was observed in one of the Cb-752 samples. Figure 16a is the metallographic appearance of the particle. Figure 16b is an absorbed electron image of the area. An electron absorption image does not identify the constituents of a sample, but is useful in showing some variations in composition and for locating microprobe images relative to visual indications. Having determined the particle to be high in silicon, oxygen, and to a lesser extent carbon, x-ray

intensity images were run for silicon and carbon as shown in Figures 16c and 16d. These images confirm the presence of these elements and indicate that they are of approximately uniform composition within the particle. A particle of this composition, essentially SiO_2, would not survive the temperature reached in melting columbium, and it is, therefore, concluded that this specific particle most likely became embedded in the sample during subsequent processing or sample preparation.

In complete metallographic examination of samples from all four alloys, the SiO_2 anomaly described was the only indication noted which could be considered as a possible inclusion type particle.

ALLOY DISTRIBUTION

X-ray intensity images were recorded for important alloy constituents for each alloy in the as-cast condition as shown in Figures 17 through 20. The x-ray intensity image is a quantitative means of determining and portraying relative concentration of one alloy constituent in different areas of a sample. In each case, the absorbed electron and x-ray intensity images are "mirror" images of the photomicrographs, which explains the lateral inversion when comparing these.

The composition of Cb-752 shows essentially no segregation of tungsten, with only a single area of possible zirconium segregation being indicated.

Some variation in tungsten may be seen in SU-31, with a very definite indication of high hafnium and low tungsten in an area of the microstructure which is heavily decorated with precipitate near a grain boundary.

The composition of C-129Y appears to be relatively uniform with the exception of minor variations of hafnium and tungsten.

As-cast C-103 shows the most distinctive segregation, there appearing to be high hafnium at the grain boundary or at apparent precipitate sites in the matrix (see Figure 20). Annealing at 2200°F does not appear to have any effect on this segregation, as shown in Figure 21. Solution treating at 2900°F (Figure 22) essentially eliminated the hafnium segregation at least as far as the detection limits of the x-ray intensity image can differentiate. As reported above, a small number of particles which are essentially hafnium oxide remain even after the 2900°F solution treatment as previously discussed in the section on individual phase analysis. These indications are consistent with metallographic examination of heat treated material.

In general, the four alloys evaluated appear to be relatively homogeneous, with the most notable exception being hafnium where it appears. Additional work will certainly be required to fully relate

the microstructure and segregation to mechanical property behavior, but information generated and reported herein establishes basic characterization of the alloys with respect to microsegregation.

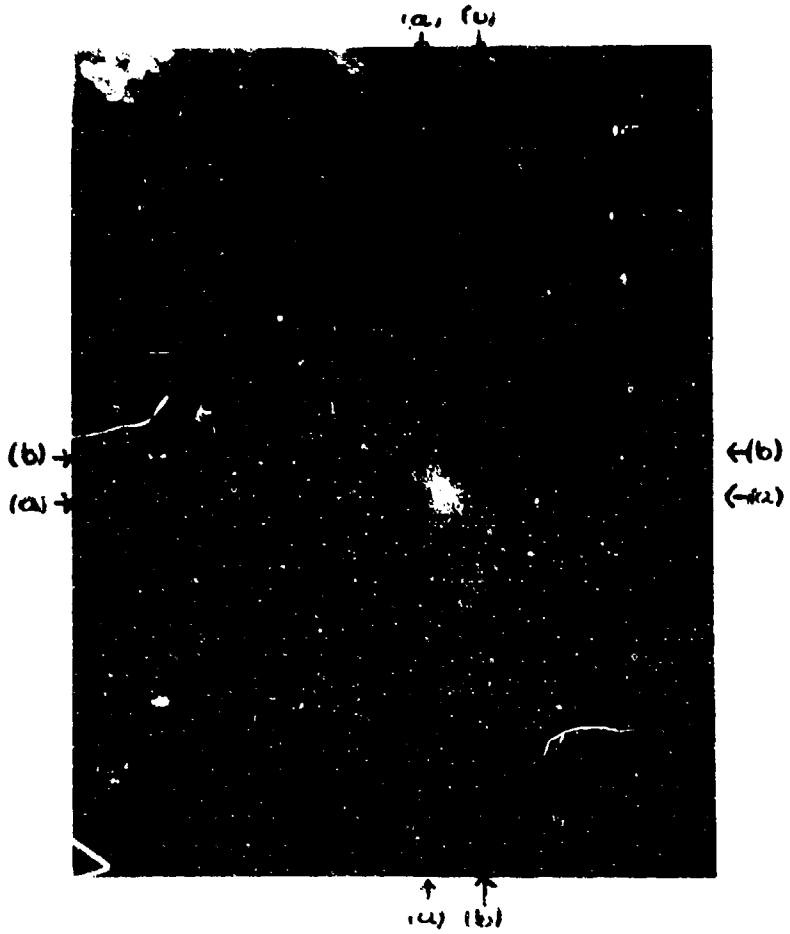
TABLE VIII

SUMMARY OF MICROPROBE TESTS

Sample #	Alloy	Heat Treat	Microprobe Targets
29331	C-103	As-Cast	Cb, Hf, Ti, Zr, Distribution
29331-1-2	C-103	2900°F/1 Hr./GC	Identify large gray particle at grain boundary
29331-1-2	C-103	2900°F/1 Hr./GC	Identify elongated particle at grain boundary
29331-1-2	C-103	2900°F/1 Hr./GC	Identify angular gray particle in matrix
29331-1-2	C-103	2900°F/1 Hr./GC	Identify elongated gray particle in matrix
29331-1-3	C-103	2900°F/1 Hr./FC	Cb, Hf, Ti, Zr, Distribution
29331-1-5	C-103	2200°F/1 Hr./FC	Cb, Hf, Ti, Zr, Distribution
29332	C-129Y	As-Cast	Cb, W, Hf, Distribution
29333	SU-31	As-Cast	Cb, W, Hf, Distribution
29334	Cb-752	As-Cast	Cb, W, Zr, Distribution
29334-4-1	Cb-752	2900°F/1 Hr./GC+ 2200°F/1 Hr./FC	Identify angular grey particle in matrix

GC = Gas cooled (Argon)

FC = Furnace cooled at 0.10 microns vacuum



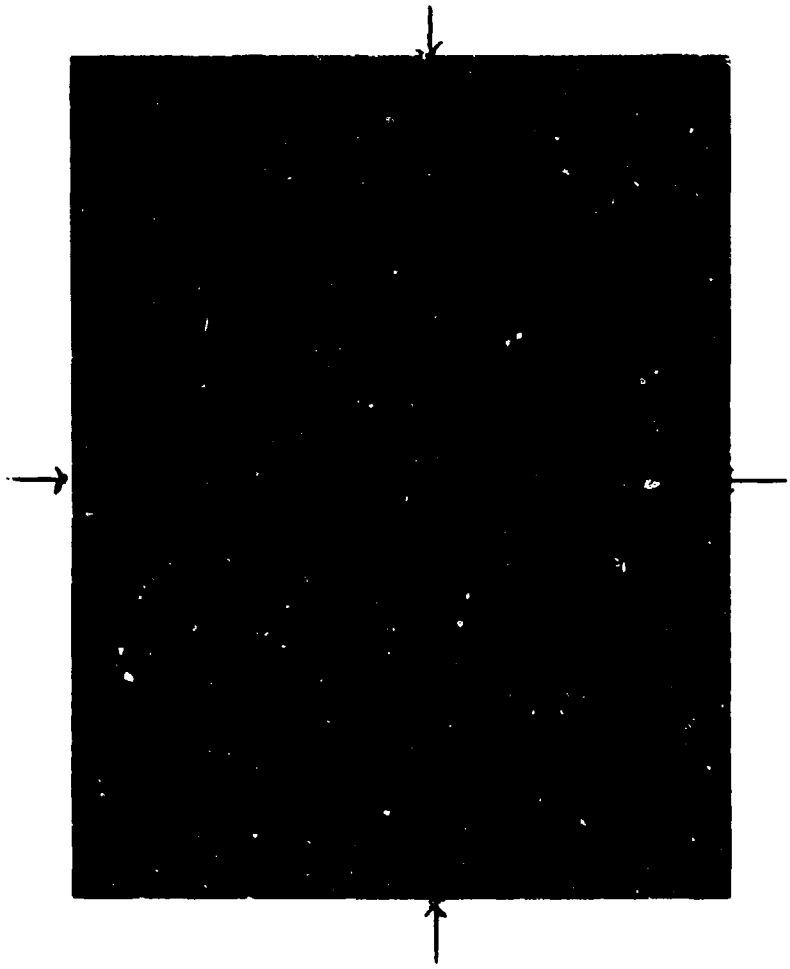
Alloy C-103
Heat Treat 2900°F/1 Hr./GC
Sample Number 29331-1-2
Area a. "Elongated Gray Particle"
Area b. "Large Gray Particle"

600X

DESCRIPTION	Cb	Hf	Ti	Zr	W	C	O	N	Y	Si
Nominal Composition	88	10	1	0.7						
Ingot Chemistry(Avg)		9.9	0.9	0.43		.0042	.0160	.0048		
Cast Chemistry						.0080	.0300	.0103		
Probe Matrix	78.3	9.85	0.90	0.36						
Probe Elong. Gray Particle at (a)	45.9	31.7	0.82	0.47		<0.5	6.50	<0.7		
Probe Large Gray Particle at (b)	11.6	58.5	0.16	0.56		1.39	24.0	<0.7		

See Table IX Note 1

Figure 13. Microprobe Analysis of C-103 Grain Boundary Particles

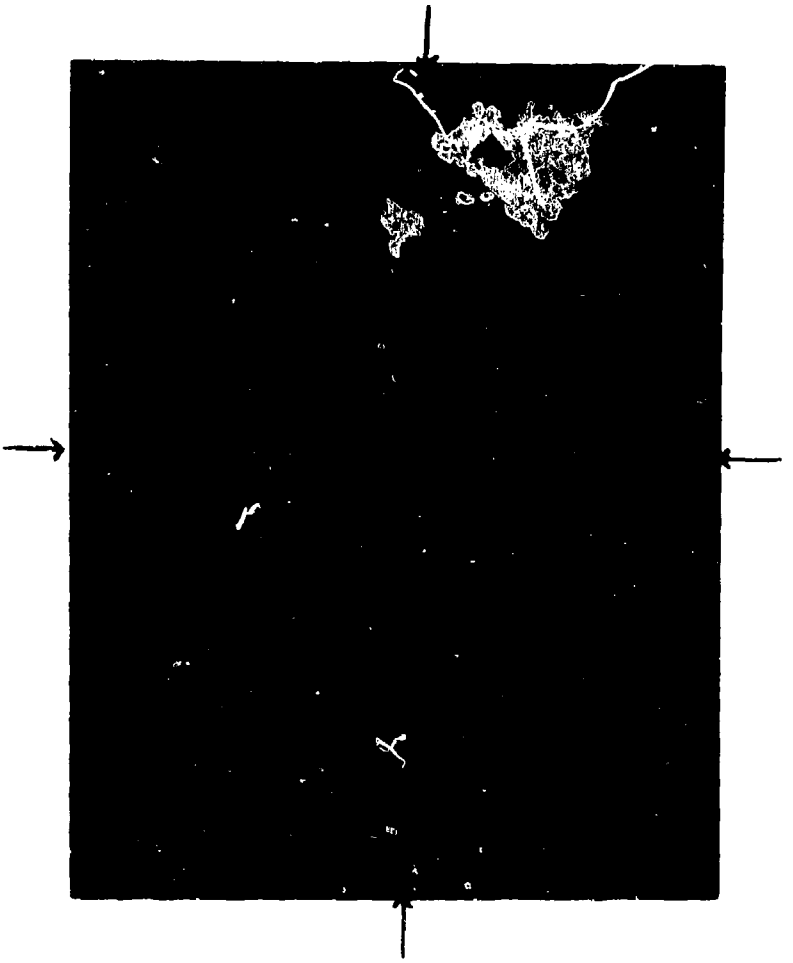


Alloy C-103 600X
Heat Treat 2900°F/1 Hr./GC
Sample Number 29331-1-2

DESCRIPTION	Cb	Hf	Ti	Zr	W	C	O	N	Y	Si
Nominal Composition	88	10	1	0.7						
Ingot Chemistry(Avg)		9.9	0.9	0.43		.0042	.0160	.0048		
Cast Chemistry						.0080	.0300	.0103		
Probe Matrix	75.8	10.9	1.02	0.39						
Probe Angular Gray Particle	5.46	69.0	0.34	0.98		1.42	23.8	<0.7		

See Table IX Note 1

Figure 14. Microprobe Analysis of C-103 Angular Matrix Particle



Alloy C-103
Heat Treat 2900°F/1 Hr./GC
Sample Number 29331-1-2

DESCRIPTION	Cb	Hf	Ti	Zr	W	C	O	N	Y	Si
Nominal Composition	88	10	1	0.7						
Ingot Chemistry(Avg)		9.9	0.9	0.43		.0042	.0160	.0048		
Cast Chemistry						.0080	.0300	.0103		
Probe Matrix	76.8	9.95	0.95	0.36						
Probe Elongated Gray Particle	26.8	49.2	0.56	0.75		<0.5	16.2	<0.7		

See Table IX Note 1

Figure 15. Microprobe Analysis of C-103 Elongated Matrix Particle

Alloy Cb-752 600X
Heat Treat 2900°F/1 Hr./GC + 2200°F/1 Hr./FC
Sample Number 29334-4-1

DESCRIPTION	Cb	Hf	Ti	Zr	W	C	O	N	Y	Si
Nominal Composition	87½			2½	10					
Ingot Chemistry(Avg)				2.7	9.7	.0075	.0145	.0042		
Cast Chemistry						.0080	.0210	.0063		
Probe Matrix	76.8			2.10	10.2					
Probe Large Angular Gray Particle	2.16			<0.17	0.38	4.38	50.4	<0.7		62.0

See Table IX Note 1

Figure 16a. Microprobe Identification of Angular Gray Particle in Matrix

Figure 16b. Absorbed electron image of area of angular gray particle. 400X.

Figure 16c. SiKα X-Ray intensity image. 400X. 1 raster traverse.

Figure 16d. CKα X-Ray intensity image. 10 raster traverses. 400X.

TABLE IX

APPROXIMATE PARTICLE COMPOSITON

Sample Number and Region of Analysis	X-Ray Intensity Ratios (1)								
	CbLα	HfLα	WLα	ZrLα	TiKα	CKα	SiKα	OKα	NKα(3)
29331-1-2, Figure 13, Matrix	78.3	9.85	N.A.(2)	0.36	0.90	Used as Background			
29331-1-2, Figure 13, Elongated Gray Particle	45.9	31.7	N.A.	0.47	0.82	Below L.D.	N.A.	6.50	Below L.D.
29331-1-2, Figure 13, Large Gray Particle	11.6	58.5	N.A.	0.56	0.16	1.39	N.A.	24.0	Below L.D.
29331-1-2, Figure 14, Matrix	75.8	10.9	N.A.	0.39	1.02	Used as Background			
29331-1-2, Figure 14, Angular Gray Particle	5.46	69.0	N.A.	0.98	0.34	1.42	N.A.	23.8	Below L.D.
29331-1-2, Figure 15, Matrix	76.8	9.95	N.A.	0.36	0.95	Used as Background			
29331-1-2, Figure 15, Elongated Gray Particle	26.8	49.2	N.A.	0.75	0.56	Below L.D.	N.A.	16.2	Below L.D.
29334-4-1, Figure 16, Matrix	76.8	N.A.	10.2	2.10	N.A.	Used as Background			
29334-4-1, Figure 16, Large Angular Gray Particle	2.16	N.A.	0.38	Below L.D.	N.A.	4.38	62.0	50.4	Below L.D.
Lower Limits of Detection	0.1	0.2	0.2	0.17	0.14	0.5	0.1	4.1	0.7
Standards Used	Pure Elements							ZrO_2	ZrN

Notes: (1) First approximations of w/o. Corrected for background, uncorrected for absorption, fluorescence and atomic number effects. Electron beam overlap of some of the smaller particles analyzed could cause the x-ray intensity ratios to vary considerably from true elemental content of the particles.

(2) N.A. = Not Analyzed

(3) Below L.D. = Below Lower Limit of Detection, if present.

DESCRIPTION	Cb	Hf	Ti	Zr	W	C	O	N	Y	Si
Nominal Composition	87½			2½	10					
Ingot Chemistry(Avg)				2.7	9.7	.0075	.0145	.0042		
Cast Chemistry						.0080	.0210	.0063		

Figure 17a. Microprobe Distribution Analysis Cb-752 As-Cast

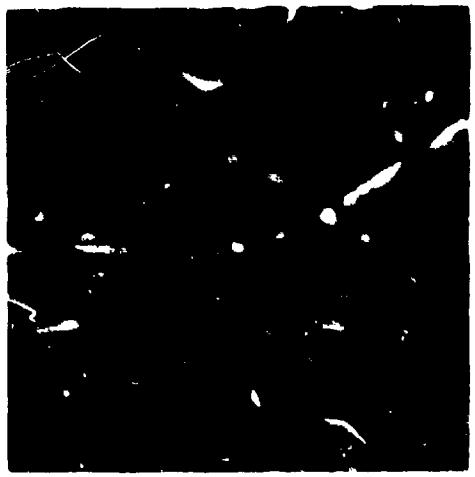


Figure 17b. Cb-752, As-Cast. Absorbed electron image of area of ZrLα examination. 1066X.

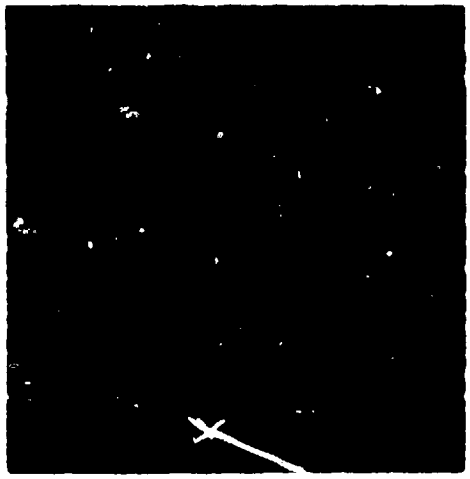


Figure 17c. Cb-752 Same area as Figure 17b. ZrLα X-ray intensity image. 5 raster traverses. 1066X.

Figure 17d. Cb-752 As-Cast. Absorbed electron image of area of CbLα and WLα examination. 1066X.

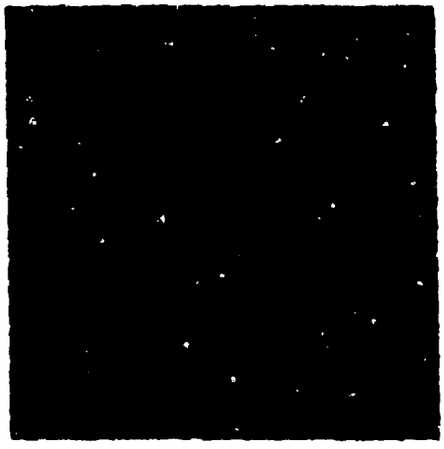


Figure 17e. Cb-752 Same area as Figure 17d CbLα X-ray intensity image. 1 raster traverse. 1066X.

Figure 17f. Cb-752 Same area as Figure 17d. WLα X-ray intensity image. 3 raster traverses. 1066X.

DESCRIPTION	Cb	Hf	Ti	Zr	W	C	O	N	Y	Si
Nominal Composition	79½	3½			17	0.12				0.03
Ingot Chemistry(Avg)		3.2			18.2	.0940	.0090	.0134		.0390
Cast Chemistry						.1010	.0050	.0070		

Figure 18a. Microprobe Distribuiion Analysis SU-31 As-Cast

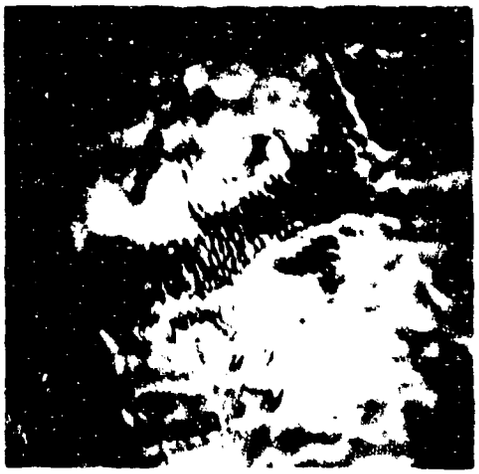


Figure 18b. SU-31, As-Cast.
Absorbed electron image.
532X.

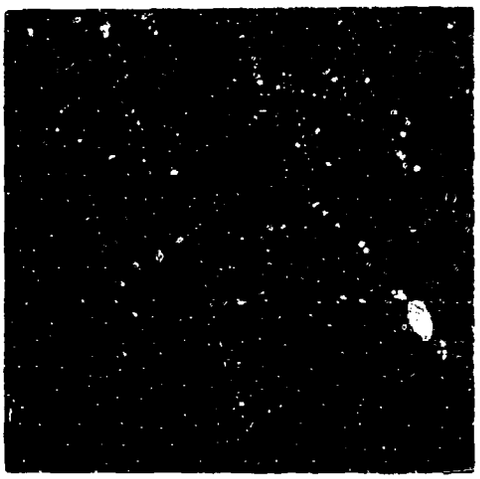


Figure 18c. SU-31
Same area as Figure 18b.
CbLα X-Ray intensity image.
532X.
1 raster traverse.

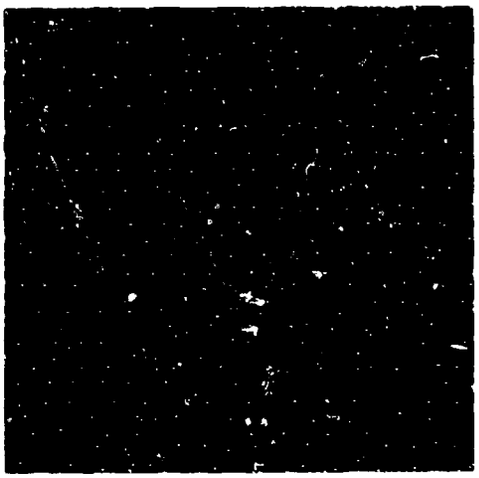


Figure 18d. SU-31, As-Cast.
Same area as Figure 18b.
HfLα X-ray intensity image.
5 raster traverses.
532X.

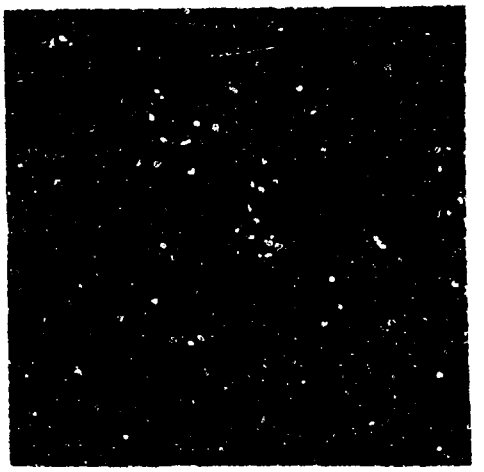


Figure 18e. SU-31, As-Cast.
Same area as Figure 18b.
WLα X-ray intensity image.
8 raster traverses.
532X.

600X

DESCRIPTION	Cb	Hf	Ti	Zr	W	C	O	N	Y	Si
Nominal Composition	80	10			10				0.1	
Ingot Chemistry(Avg)		8.8			9.6	.0060	.0050	.0038	0.07	
Cast Chemistry						.0040	.0110	.0038		

Figure 19a. Microprobe Distribution Analysis C-129Y As-Cast

Figure 19b. C-129Y, As-Cast. Absorbed electron image of area of CbLα and HfLα examination. 1066X.

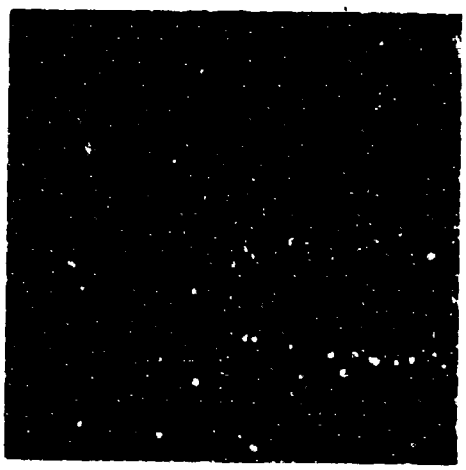


Figure 19c. C-129Y, Same area as Figure 19b. CbLα X-ray intensity image. 1 raster traverse. 1066X.

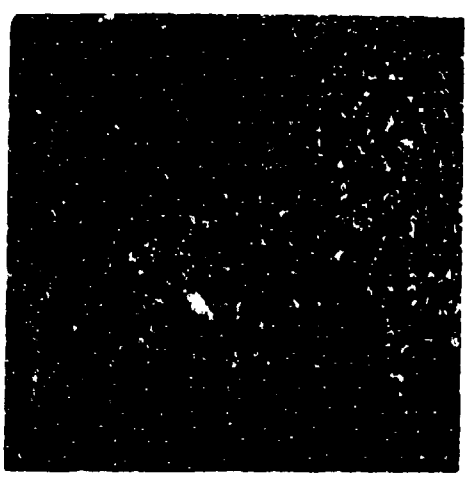


Figure 19d. C-129Y, Same area as Figure 19b. HfLα X-ray intensity image. 3 raster traverses. 1066X.

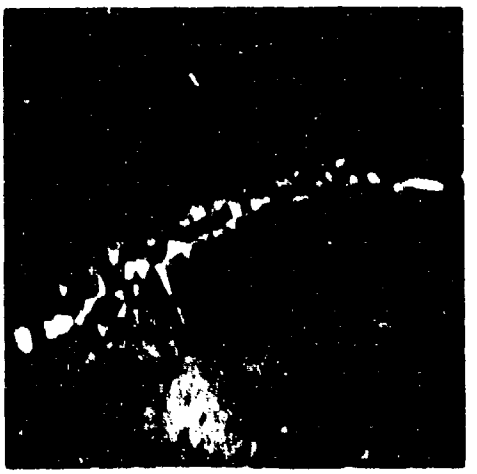


Figure 19e. C-129Y, As-Cast. Absorbed electron image of area of WLα and YLα examination. 1066X.

Figure 19f. C-129Y, Same area as Figure 19e. WLα X-ray intensity image. 5 raster traverses. 1066X.

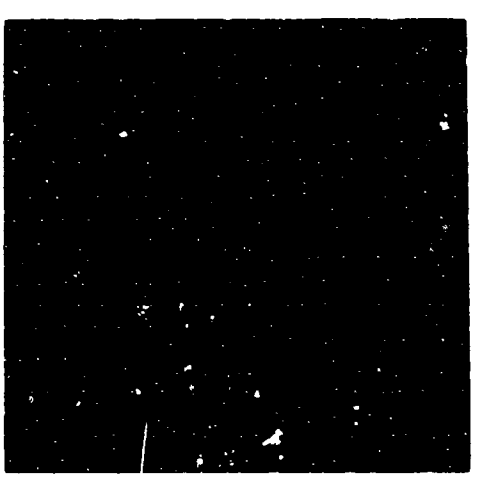


Figure 19g. C-129Y, Same area as Figure 19e. YLα X-ray intensity image. 1066X.

DESCRIPTION	Cb	Hf	Ti	Zr	W	C	O	N	Y	Si
Nominal Composition	88	10	1							
Ingot Chemistry(Avg)		9.9	0.9	0.43		.0042	.0160	.0048		
Cast Chemistry						.0080	.0300	.0103		

Figure 20a. Microprobe Distribution Analysis C-103 As-Cast

Figure 20b. C-103, As-Cast. Absorbed electron image of area of CbLα and HfLα examination. 1066X.

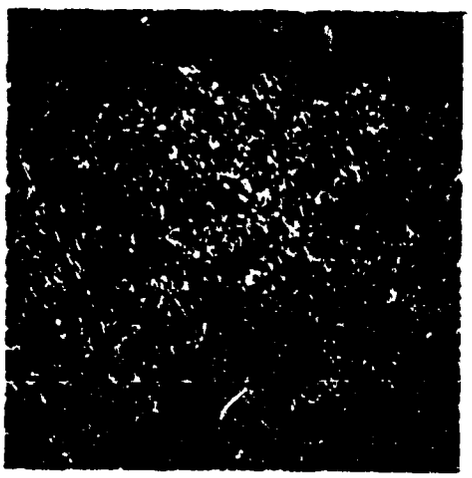


Figure 20c. C-103, Same area as Figure 20b. CbLα X-ray intensity image. 3 raster traverses. 1066X.

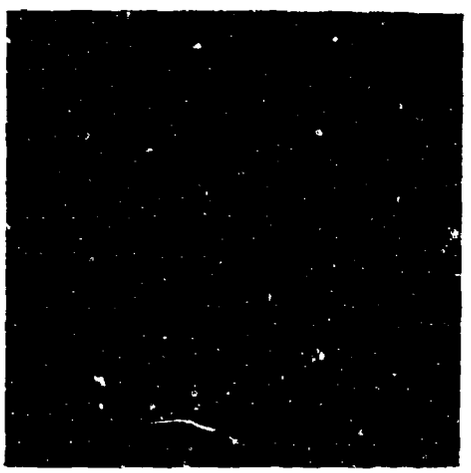


Figure 20d. C-103, Same area as 20b. HfLα X-Ray intensity image. 3 raster traverses. 1066X.

Figure 20e. C-103, As-Cast. Absorbed electron image of area of ZrLα and TiKα examination. 1066X.

Figure 20f. C-103, Same area as Figure 20e. ZrLα X-ray intensity image. 5 raster traverses. 1066X.

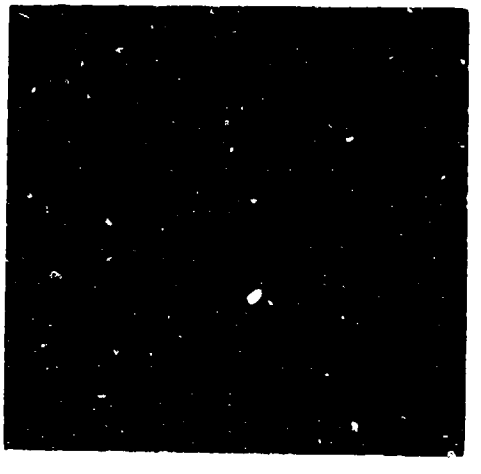
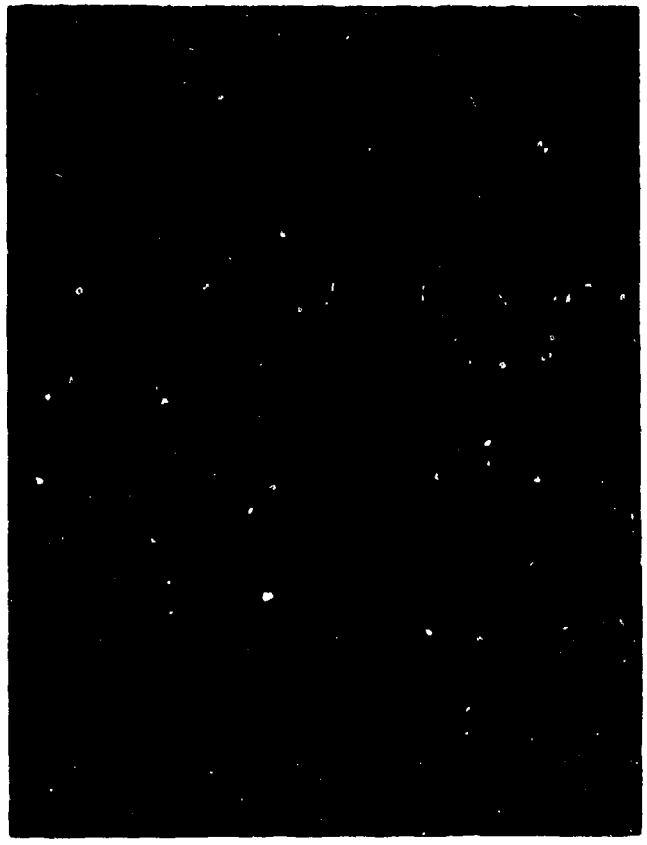


Figure 20g. C-103, Same area as Figure 20e. TiKα X-ray intensity image. 5 raster traverses. 1066X.



600X

DESCRIPTION	Cb	Hf	Ti	Zr	W	C	O	N	Y	Si
Nominal Composition	88	10	1							
Ingot Chemistry(Avg)		9.9	0.9	0.43		.0042	.0160	.0048		
Cast Chemistry						.0080	.0300	.0103		

Figure 21a. Microprobe Distribution Analysis C-103 Annealed

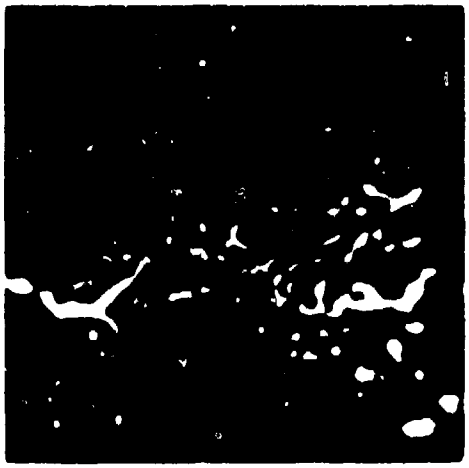


Figure 21b. C-103, Absorbed electron image.
1066X.

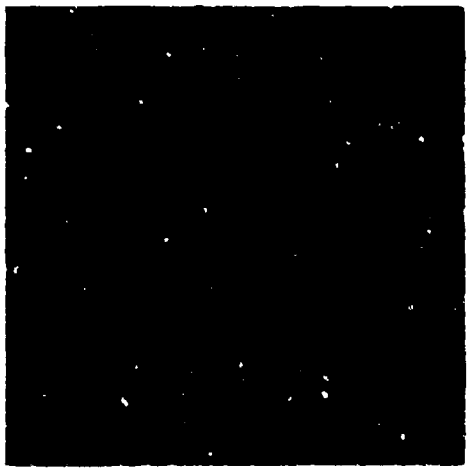


Figure 21c. C-103, Same area as Figure 21b. CbLα X-ray intensity image.
2 raster traverses.
1066X.

Figure 21d. C-103. Same area as Figure 21c. HfLα X-ray intensity image.
5 raster traverses.
1066X.

Figure 21e. C-103, Same area as Figure 21b. ZrLα X-ray intensity image. 5 raster traverses. 1066X.

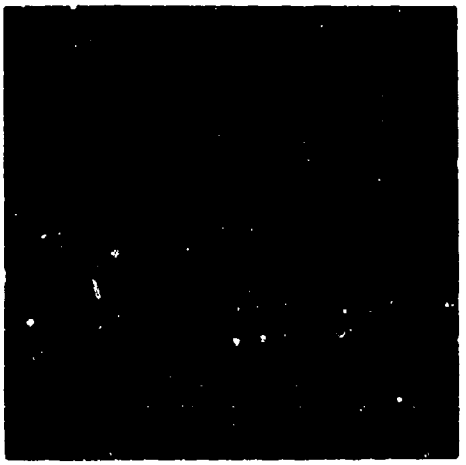


Figure 21f. C-103, Same area as Figure 21b. TiKα X-ray intensity image. 5 raster traverses. 1066X.

600X

DESCRIPTION	Cb	Hf	Ti	Zr	W	C	O	N	Y	Si
Nominal Composition	88	10	1							
Ingot Chemistry(Avg)		9.9	0.9	0.43		.0042	.0160	.0048		
Cast Chemistry						.0080	.0300	.0103		

Figure 22a. Microprobe Distribution Analysis C-103 Solution Treated

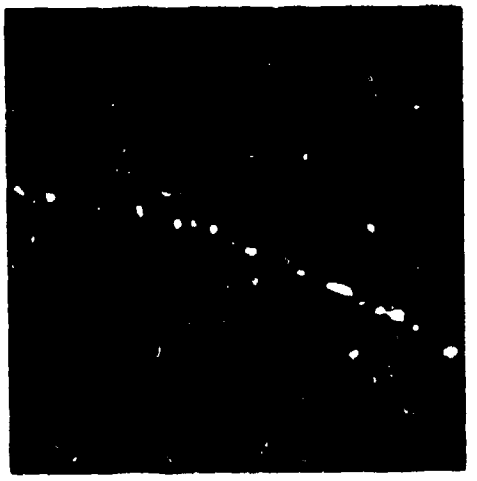


Figure 22b. C-103, Absorbed electron image of area of CbLα and HfLα examination. 1066X.

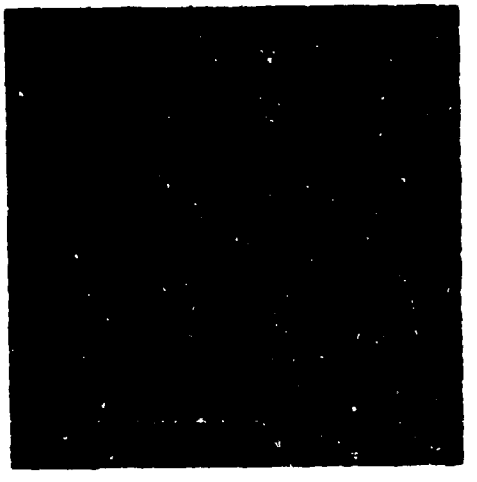


Figure 22c. C-103, Same area as Figure 22b. CbLα X-ray intensity image. 1 raster traverse. 1066X.

Figure 22d. C-103, Same area as Figure 22b. HfLα X-ray intensity image. 5 raster traverses. 1066X.

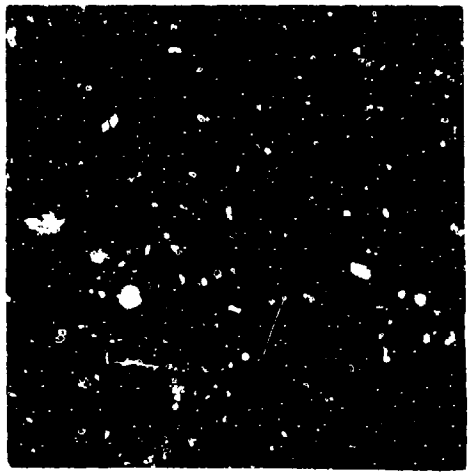


Figure 22e. C-103, Absorbed electron image of area of ZrLα and TiKα examination. 1066X.

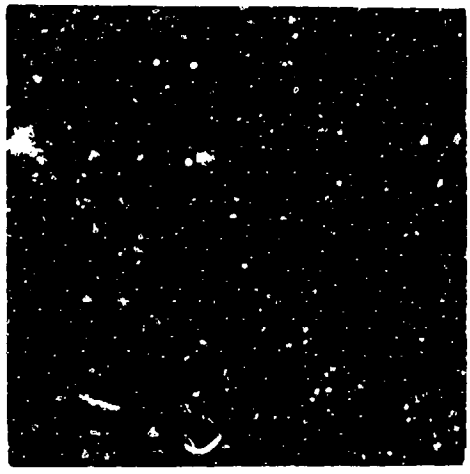


Figure 22f. C-103, Same area as Figure 22e. ZrLα X-ray intensity image. 5 raster traverses. 1066X.

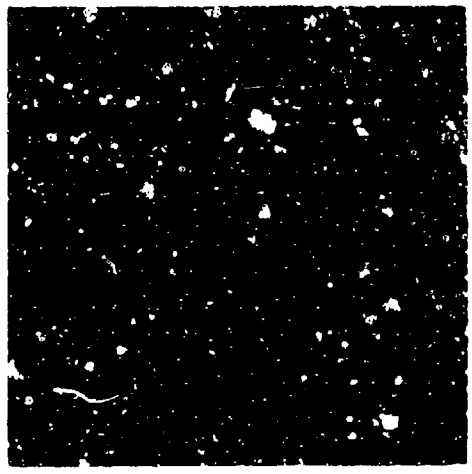


Figure 22g. C-103. Same area as Figure 22e. TiKα X-ray intensity image. 5 raster traverses. 1066X.

SECTION VII

MECHANICAL PROPERTIES FOR ALLOY COMPARISON

Room temperature and elevated temperature tensile properties were determined on castings of each of the four alloys. Standard round specimens as shown in Appendix 3 were machined from the cast blanks. All specimens were uncoated. Room temperature tests were performed in air, while elevated tests were carried out either in vacuum or in an atmosphere of high pruity argon.

The tensile results obtained from the alloy comparison tests are tabulated in Appendix 2 along with additional confirming tests run on C-103 and NDT characterization of the specimens.

The stress-strain curves for each alloy at room temperature are shown in Figure 23. The modulus of elasticity calculated from each room temperature test is reported in Table X. These values represent reasonably good agreement with typical mill product properties for the alloys.

The elevated temperature stress-strain curves were recorded in two ways, as shown and described in Figure 24. For C-103 three separate tests were performed. The first was run basically to qualify the technique and was performed prior to decision to actually calculate the yield strength by both of the possible techniques. This specimen was analyzed for oxygen and it was determined that no significant contamination of the specimen occurred as a result of the test. The second specimen was run for verification and calculation of the yield strength both ways. The third specimen was submitted to Wah Chang for a tensile test in vacuum according to their normal methods. The lower overall strength and ductility of the third specimen is primarily attributable to a defect in the third specimen rather than to a difference in testing method. For each of the other alloys, a single elevated test was run for comparison to the other alloys and to the wrought typical values.

Some difficulty was encountered in recording the continuously constant actual loading rate and there are certain features of the curves which are not fully explainable. The curves were considered adequate for determination of the yield strength, but did not produce meaningful elastic modulus data.

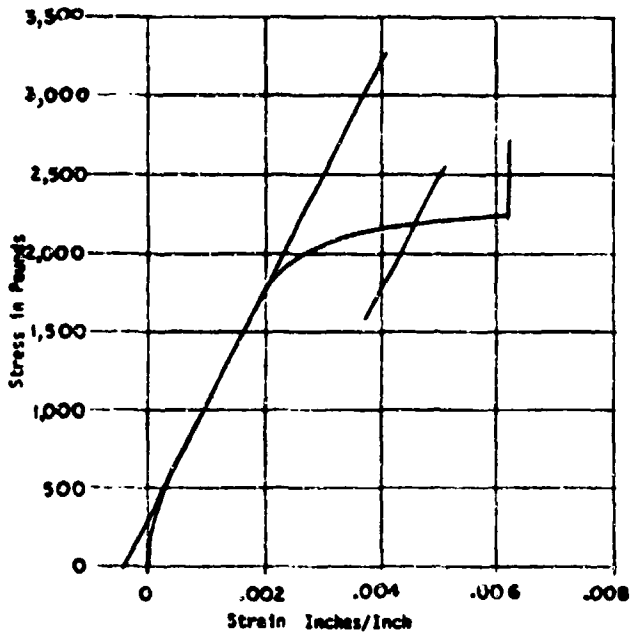
Figure 25 is a graphic portrayal of the comparison between cast and wrought tensile properties for the four alloys. It is not possible from the tests run to make any real interpolation of properties between room temperature and 2200°F for the cast material, and the curves shown in Figure 25 are for illustration of the actual data only.

In the room temperature tests, SU-31 confirms the brittle behavior experienced with this alloy in previous evaluations, being considerably below the five-percent elongation guideline The best

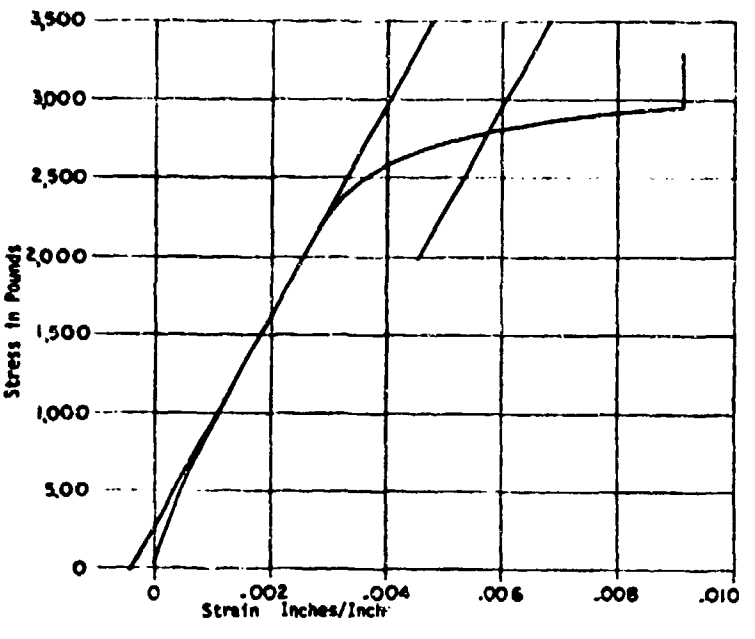
ductility was observed with C-103, which incidentally was also the only alloy to surpass typical wrought room temperature strength. Ductility of Cb-752 and C-129Y are marginal with respect to the five-percent minimum elongation. Values for Cb-752 are much lower than some pre-program results on this alloy. an occurrence which is tentatively attributed to a greater dependence on section size and resulting grain size and structure.

In general, the elevated temperature strength levels agree fairly well with the wrought typical values. Ductility was much lower than the comparable wrought values in all cases. Two of the alloys, SU-31 and Cb-752, did not meet the program goal minimum of five-percent elongation in one or more tests although Cb-752 was quite close. Ductility of the other two alloys was lower than would be predicted from room temperature data, but was acceptable with respect to the five-percent minimum. It is possible that one or more of the tests was affected by interstitial contamination, but probably not sufficiently to change any conclusion which would be reached. Since the number of tests scheduled and carried out was rather limited, it is difficult to place much significant on the exact numbers obtained.

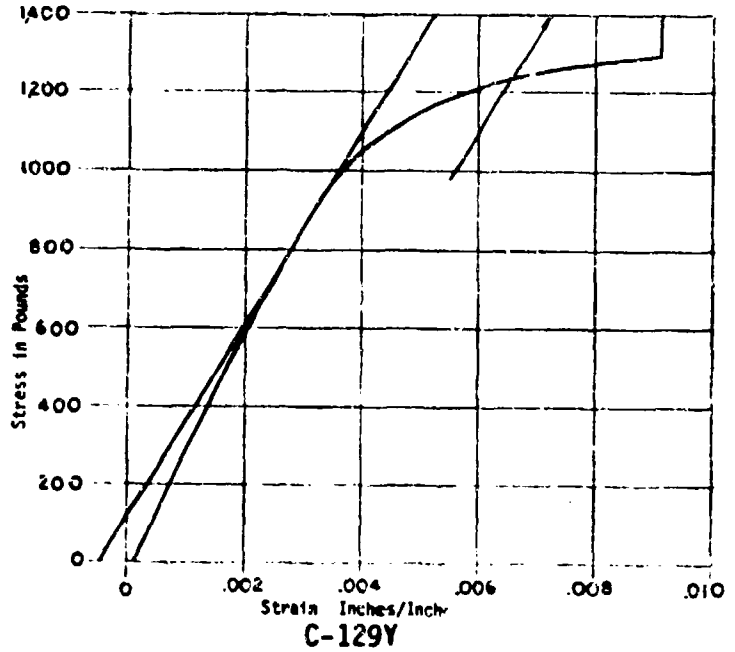
However, it is apparent that the properties generally follow the same relationships of strength and ductility as was observed at room temperature. One alloy, SU-31, would be disqualified at least in the as-cast condition, due to poor ductility. The second strongest alloy, C-129Y, would appear to be the best from strictly a tensile property standpoint based on the single test.



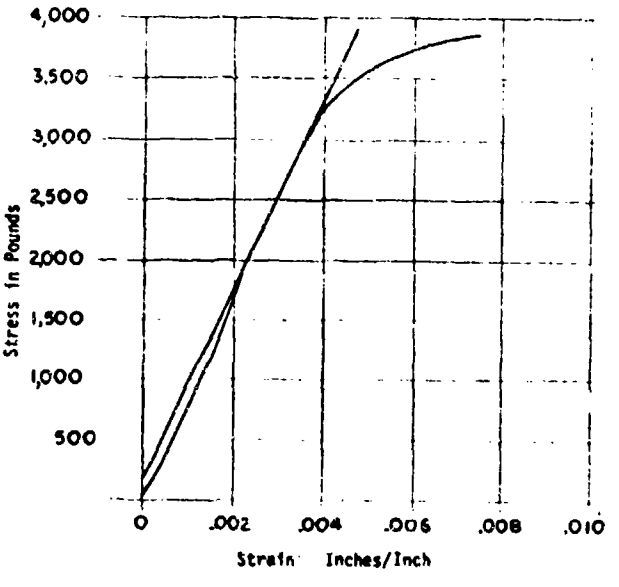
C-103



Cb-752



C-129Y



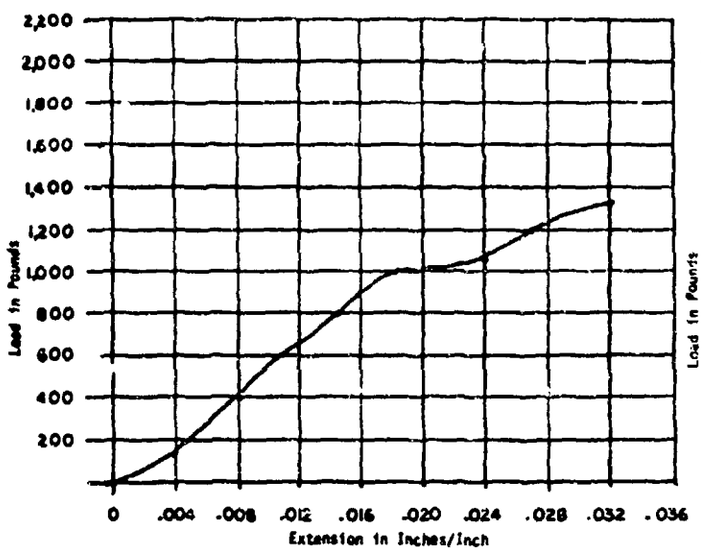
SU-31

Figure 23. Room Temperature Stress-Strain Curves

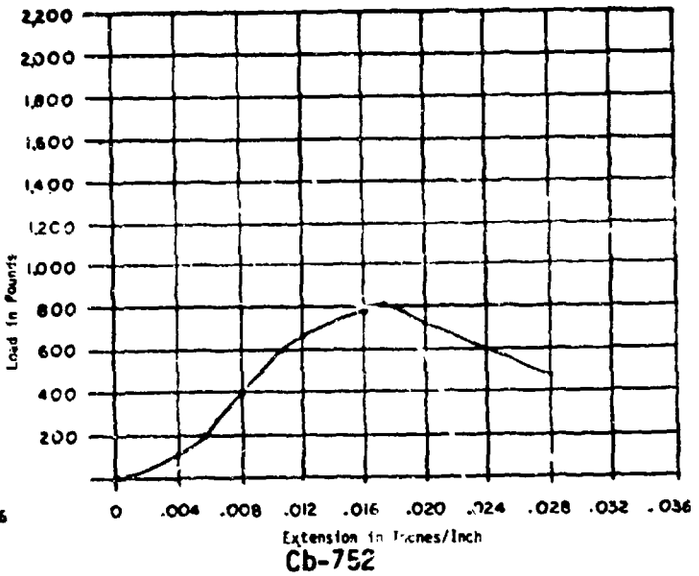
TABLE X

ROOM TEMPERATURE MODULII OF ELASTICITY

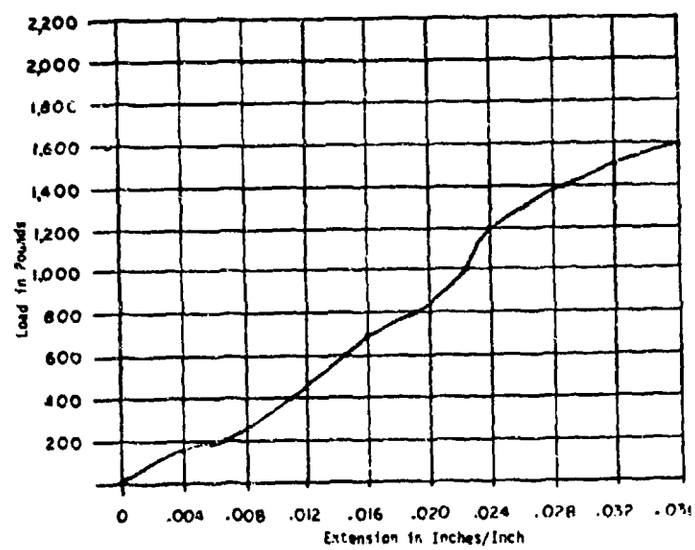
	C-103				Cb-752				C-129Y				SU-31			
	Test 1	Test 2	Ave.	Typ. Wrt.	Test 1	Test 2	Ave.	Typ. Wrt.	Test 1	Test 2	Ave.	Typ. Wrt.	Test 1	Test 2	Ave.	Typ. Wrt.
E X 10^{-6}	15.15	15.67	15.42	13.1	14.1	15.70	14.9	15.03	14.90	16.51	15.71	16.3	16.51	20.40	18.46	17.7
Test Bar No.	7156	7150			7144	7146			7164	7167			7137	7136		



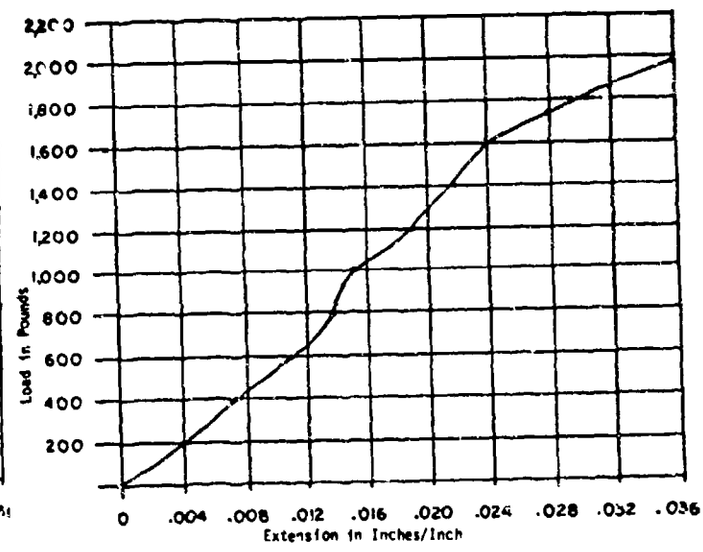
C-103



Cb-752

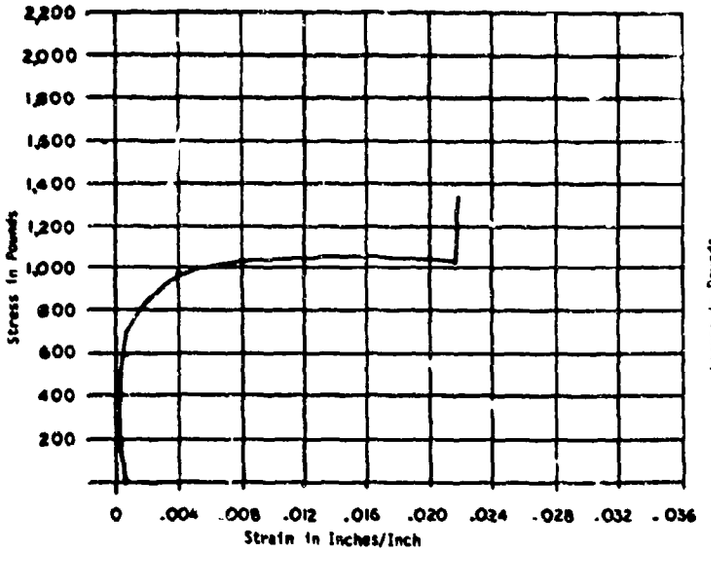


C-129Y

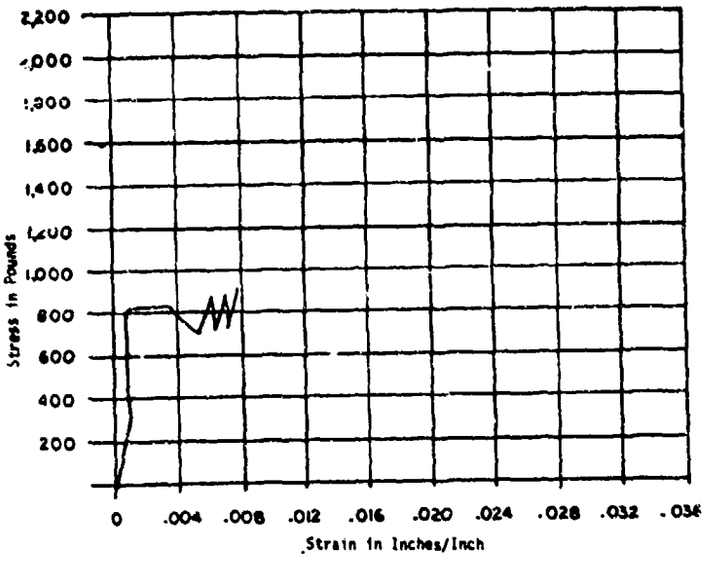


SU-31

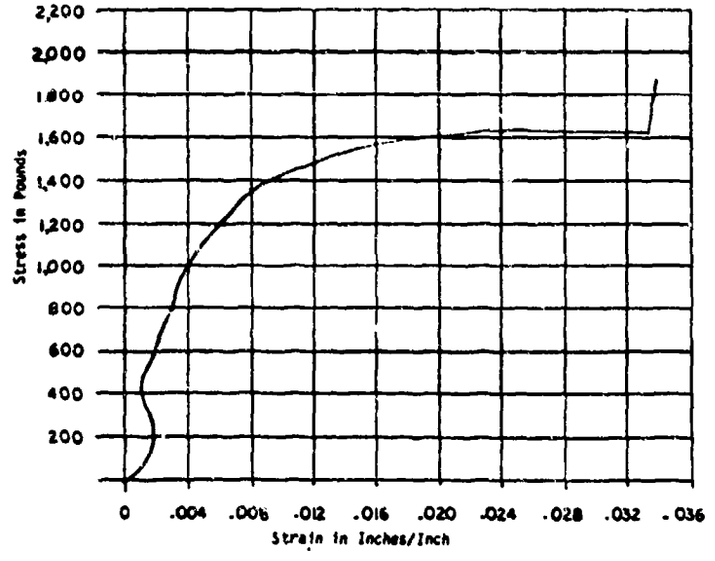
Figure 24a. 2200°F Load Extension Curves Calculated From Cross Head Movement and Actual Gage Elongation



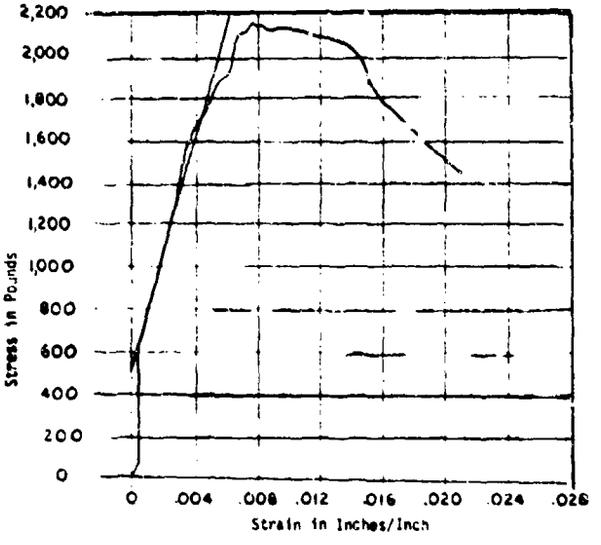
C-103



Cb-752

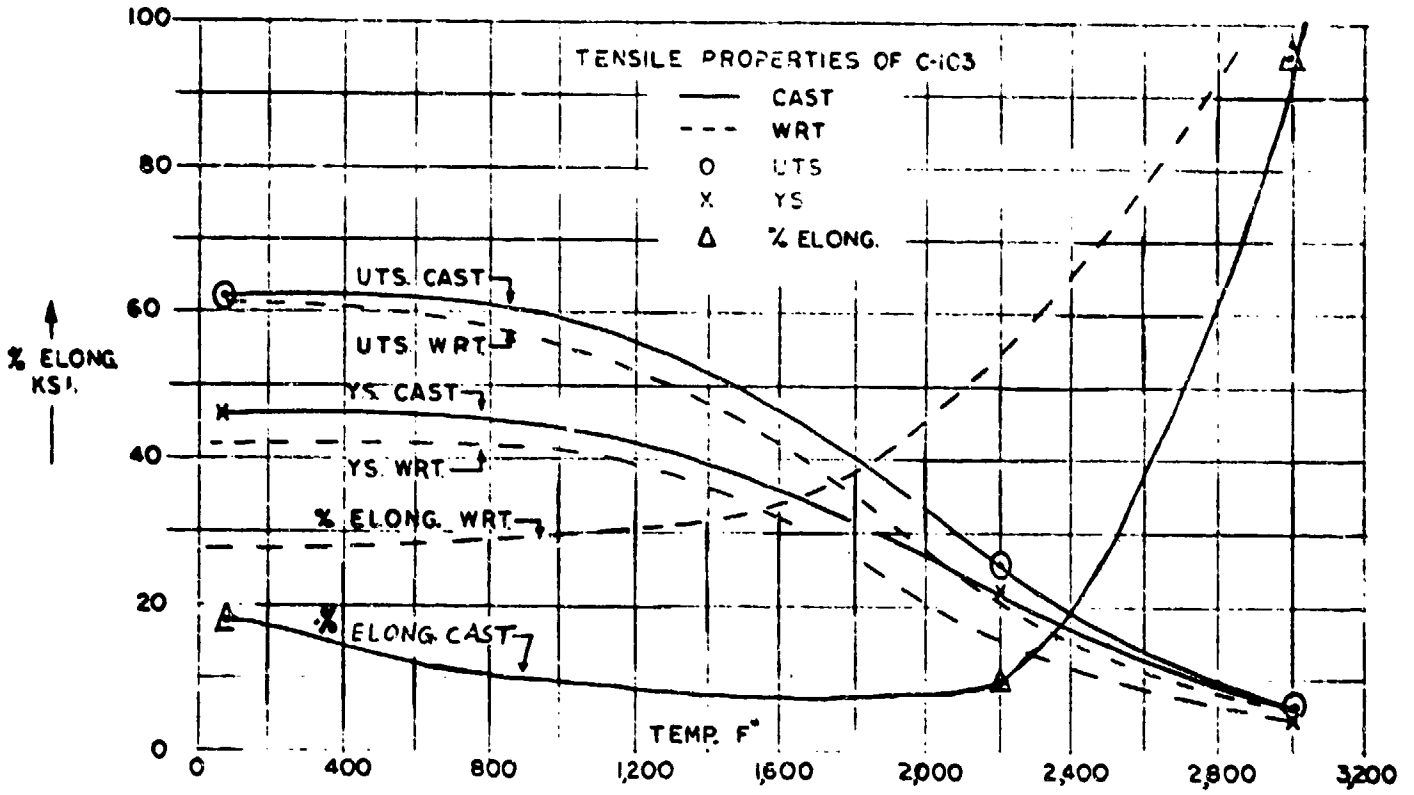


C-129Y



SU-31

Figure 24b. 2200°F Stress-Strain Curves
Calculated by Extensometer Movement



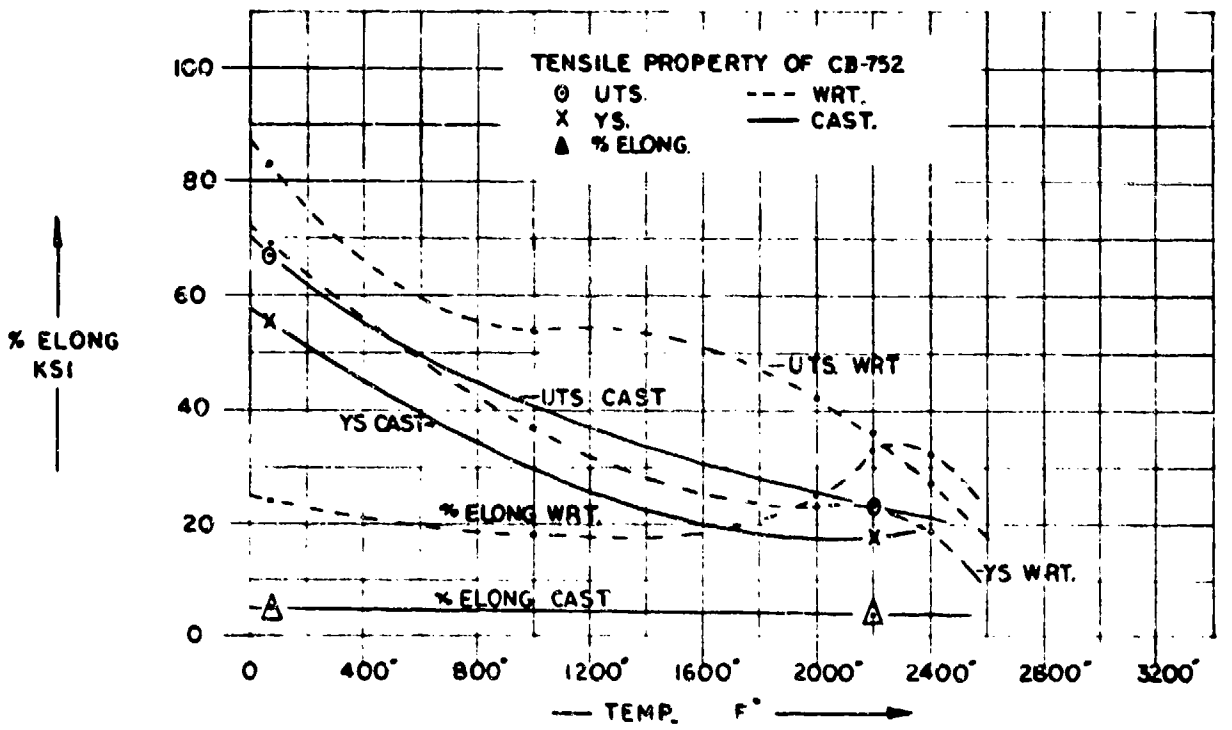
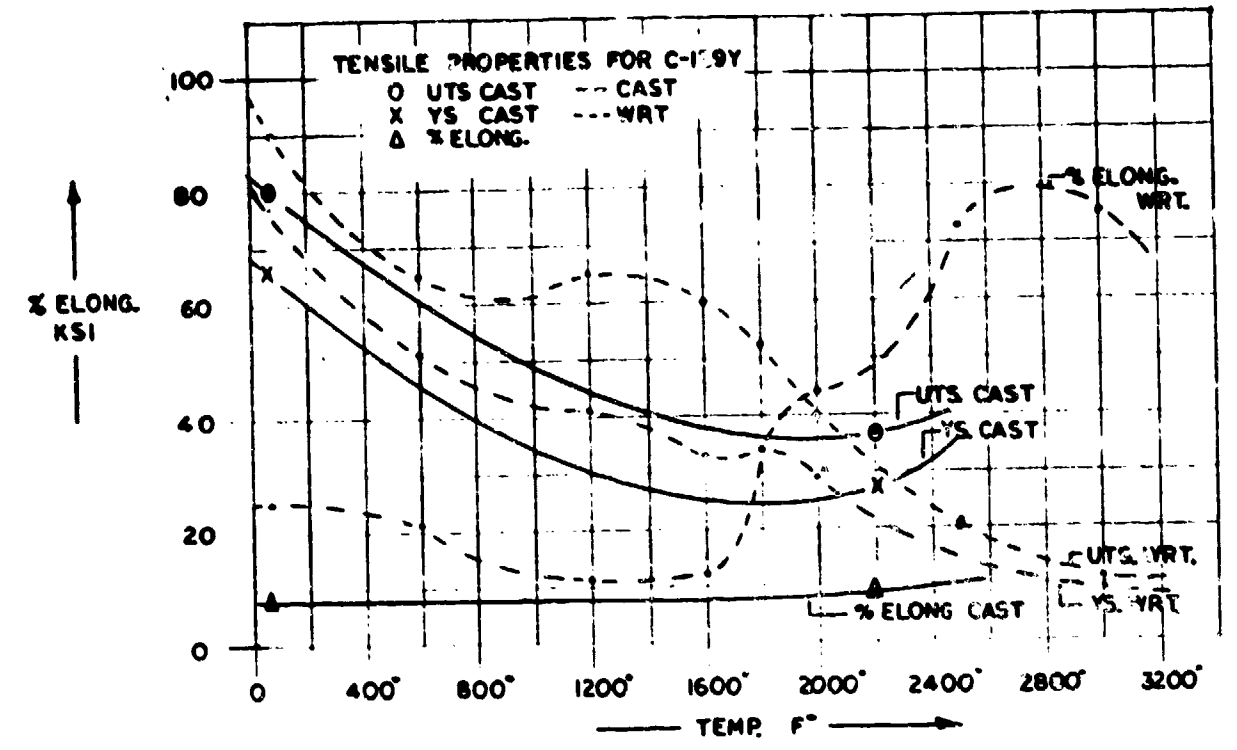


Figure 25. Elevated Temperature Tensile Properties



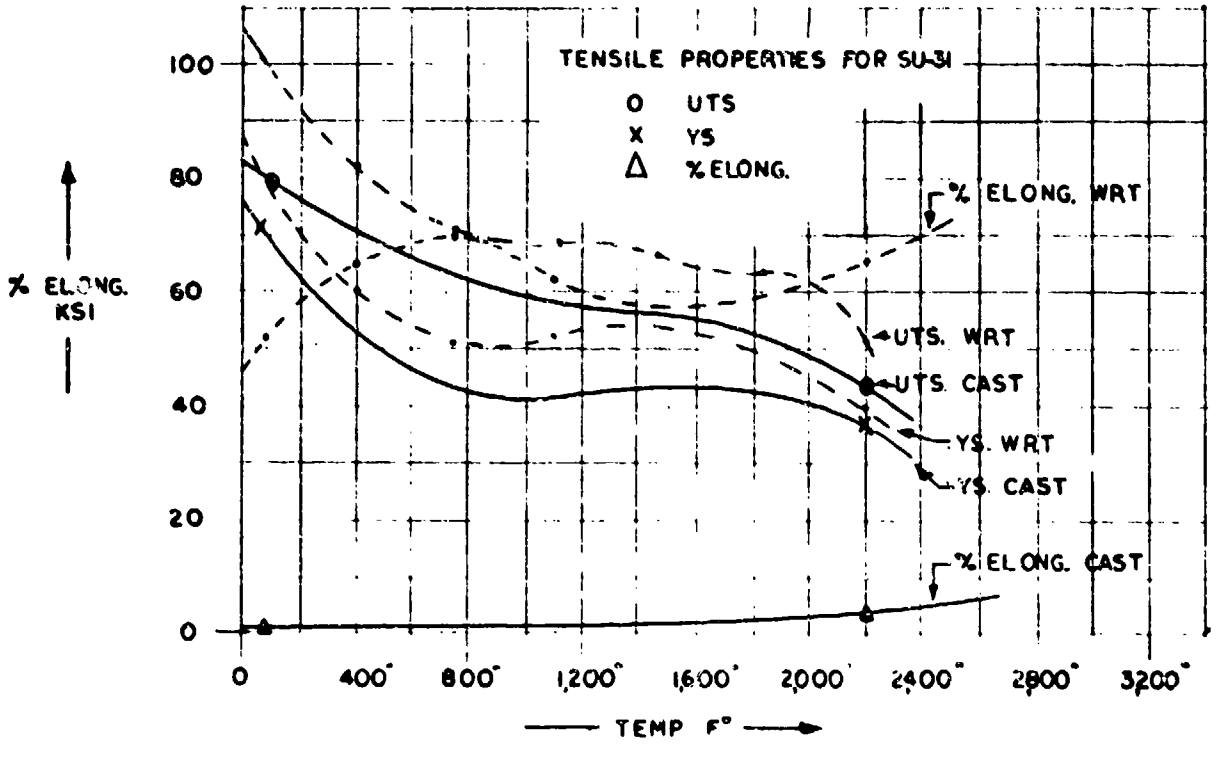


Figure 25. (Cont'd.) Elevated Temperature Tensile Properties

SECTION VIII

ALLOY EVALUATION AND SELECTION

In this section, the criteria for selection of a columbium alloy will be discussed and applied to the information available on each of the four cast alloys under investigation.

1. Room Temperature Tensile Properties

One of the ground rules of this program are minimum tensile properties as follows:

Ultimate Tensile Strength	40 KSI
Yield Strength	36 KSI
Elongation	5%

One of the alloys, SU-31, failed to meet the five-percent elongation. Elongation of C-103 met the five-percent minimum with a very good margin. The other two alloys demonstrated that they could meet the limit but with a much smaller margin. All of the four alloys readily met the strength requirements. Based on these room temperature tests, C-103 would be selected for further investigation or use.

2. Elevated Temperature Tensile Properties

The program property goals are as follows:

	1800°F	2200°F	2500°F
Ultimate Tensile Strength	27.5 KSI	20.0 KSI	12.0 KSI
Yield Strength	23.5 KSI	16.5 KSI	9.6 KSI
Elongation	5%	5%	5%

Each of the alloys demonstrated acceptable strength levels, although C-103 did not do so in all tests. Ductility of C-103 and C-129Y met the five-percent minimum limitation. Based upon the single elevated temperature tests alone, selection would be limited to C-103 and C-129Y.

3. Density

In the absence of all other considerations, the lightest alloy, C-103, would be selected. (Note: Due to limitations in minimum section thickness useable or producible, the density of those alloys with adequate properties could be a more important criterion than strength to weight ratio.)

4. Strength to Weight Ratio

Room temperature strength to weight ratio indicates that C-129Y would be the best selection:

	C-103	Cb-752	C-129Y	SU-31
Density g/cc	8.85	9.02	9.49	9.50
Ultimate Strength to Weight Ratio KSI/gm/cc	6.98	7.47	8.43	7.92

5. Thick Versus Thin Section Properties

Any conclusion here is tentative and based on very limited and not entirely comparable information. The program data coupled with previous data indicates that C-103 and SU-31 are relatively insensitive to section thickness, whereas Cb-752 appears to be very sensitive; and there are no adequate data for a conclusion on C-129Y.

6. Modulus of Elasticity

In the absence of very specific design requirements, as well as statistical analysis of a greater number of test results, it is not possible to differentiate between the alloys or select one with the most desirable modulus.

7. Impact Resistance (Casting Data Not Available)

Very good charpy impact values on the order of 150 ft.-lbs. were found only for wrought C-103. Impact bending data were found for C-129Y and Cb-752, but are not directly comparable with the charpy test. SU-31, with a low ductility, would not be expected to have very good impact resistance, at least at low temperatures.

8. Oxidation Resistance

None of the alloys is considered particularly oxidation resistant.

9. Heat Treat Response

SU-31 was originally considered most promising from a heat treat response standpoint. A much better microstructural response was demonstrated with C-103 than with SU-31, although it is felt that different parameters would ultimately prove more beneficial to the properties of SU-31.

10. Weldability

In general, alloys with better ductility are considered more weldable in the absence of complicating factors. There is no definite basis for predicting any other order of preference than that shown for room temperature ductility.

11. Machinability

Machinability can be either a positive or negative factor in selection of a cast alloy which could also be used as a machined forging or mill product. Finish machining of the casting requires acceptable machinability, whereas typically the economic benefits of using a casting are more prevalent in difficult-to-machine materials. Columbium is in general a fairly difficult-

to-machine material type. The higher hardness of SU-31, relative to the others, would be expected to reduce galling which is a major problem in machining columbium.

12. Castability

Unusual difficulty was experienced only with one alloy, C-129Y. The problem with this alloy appears to be related to the yttrium content and does not appear insurmountable. However, it is a significant deterrent to this alloy relative to the others. With respect to other factors involved in castability, it has not been established that any one of the alloys is better than another for fluidity, cold shut, cold lap, or centerline shrinkage.

13. Cost and Availability

All of the alloys except SU-31 are produced by more than one source. In the case of SU-31, the situation is rather restrictive, and the alloy is available domestically from only one source. Partially due to greater difficulty in handling the alloy, and presumably also to some extent due to the existence of only a single source, SU-31 is substantially higher in price than the other alloys. This difference, however, is expected to diminish as greater quantities are produced. Looking ahead to the potential quantities of columbium alloys which could be used, the relatively limited availability of Hafnium could potentially affect availability or cost of C-129Y, SU-31, and C-103. If Hafnium availability were to be a limiting factor, then Cb-752 would be favored since it has no Hafnium requirement. At the present time, however, this does not appear to be the controlling factor.

As of May, 1975, the approximate relationship of prices of electrode stock for the four alloys is as follows (quantities approximately 500 pounds):

C-129Y	$48.50
C-103	$40.00
Cb-752	$40.00
SU-31	$59.62

14. Scrap Value and Recovery

At the present time, there is essentially no difference in the value of casting scrap from the four alloys. This value, for anything except "foundry return" use, is on the order of only two to four percent of the original material value. When used as "foundry returns", that is, remelted directly in the production of new castings, the material is nearly as valuable as original electrode stock, differing only in the cost of additional cleaning and electrode fabrication. Alloy composition will have an influence on the effect of interstitial impurity changes. Since

interstitials introduced into the metal through multiple recycles will most drastically affect ductility, the alloy with the best ductility, C-103, would be selected based on real scrap recovery value.

CONCLUSION

In selection of an alloy for additional program work, or for any application, it is clear that any choice will not be best in all categories. C-103 was the alloy selected for additional investigation and for production of the vane castings in this program. The reasons for this selection may be summarized as follows:

1. The alloy meets the minimum requirements for strength and operating temperature capability.

2. It excells the other alloys in ductility and related behavior, such as weldability and recycleability potentials.

3. Over the short term, the alloy is the most readily available and the least costly.

4. The alloy has the lowest density while still meeting property minimums. Assuming a practical limitation on minimum producible wall thickness, C-103 would produce the lightest component.

SECTION IX

CASTING OF AIRFOILS

As part of this advancement of the state of the art in columbium castings, the process capability was applied to an airfoil configuration having characteristics which would typically be found in a pratical vane for turbine engine use.

The airfoil contour itself was taken from a glass layout of a vane previously produced in another material. A simple contour was used rather than a compounded contour configuration having curvature both from leading to trailing edge and from platform to platform. This was done for economies in the tooling and does not represent a limitation of the process.

The main reason for internal coring in conventional turbine vane materials is for a passage for cooling air to increase the range of gas temperatures in which the vane can function. Some engine efficiency is lost due to use of cooling air. The projected real operating temperature capabilities of columbium alloys indicate that the use of cooling air will likely not be necessary. Therefore, the use of a hollow vane rather than a solid vane will probably not be essential.

For the columbium components, however, there are some other reasons why a cored configuration should be retained. First, where strength is adequate, a cored part will be lighter than a solid part of the same size. Second, a cored structure having relatively thin walls may be less susceptible to cyclic stress or thermal fatigue than the comparable solid part. There are other factors which will also have some influence such as the fact that very complex cores such as are used in many vane designs can neither be produced in columbium nor successfully coated.

These factors must obviously be considered for optimization of specific designs. Since there may be practical useage of both cored and solid vanes, the configuration chosen for this program included both options.

The drawing for the airfoil is shown along with the test specimen drawings in Appendix 3. Figure 26 shows the wax injection molding tooling, disassembled to show the internal configuration. The internal core configuration is formed using a core rod of the same contour as the outside except smaller. One of these core rods was sized such that a 0.060 inch wall thickness was produced, shown in the lower right attached to a face plate. A second core rod was sized to give a 0.040 inch wall and is shown alone near the center of the picture. Selection of the exact wall thickness values 0.040 inch and 0.060 inch were based on several considerations. Most importantly, the 0.060 inch thickness was considered reasonably likely to be achieved based on existing knowledge of fluidity and feeding

characteristics. The thinner 0.040 inch was considered a good target for extension of the state of the art, although probability of achievement within the current program was not considered to be high.

Figure 27 shows wax patterns of the airfoil configuration having different cores or no core at all. Patterns for solid vanes were prepared by filling the as-molded pattern with additional wax.

Figure 28 represents four views of one of the first two similar molds prepared to determine filling and feeding characteristics of the three modifications of airfoils. As can be seen, some test specimens also are included in the set-up. Each mold contained three solid airfoils, three airfoils with 0.060 inch walls, and two airfoils with 0.040 inch walls. Molds were prepared from the pattern assemblies using normal procedures. Foundry parameters are shown in Table XI for these and the remaining airfoil castings pours.

The resulting castings are shown in Figures 29 and 30 still on the sprue. NDT results beginning with visual examination of degree of fill are reported in Section X. Based upon the degree of fill obtained, the gating configurations which performed best were selected for use in additional pours. The mold temperature was found not to have a significantly favorable effect above 1000^{0}F, and therefore, 1000^{0}F was chosen for the balance of the pours.

It is worthy of note that the first vane pour, Lot J was made using an electrode which was actually assembled using a large amount of revert from the original test specimen pour. The little change in chemistry or hardness as shown in the appendices indicate that recycling of the material with adequate intermediate cleaning, processing, and inspection is expected to be extremely practical.

Following evaluation of the first molds, four additional molds were prepared and cast. The wax pattern assemblies and partially cleaned castings, prior to cut-off are shown in Figures 31 and 32 respectively.

The airfoil castings were cut off and finish-cleaned according to normal procedures without any unusual problems, with the notable exception of the trailing edge of the internal core section. In the mold, this area is a very thin tungsten structure, being as thin as 0.025 inch. Due to the high degree of retained heat and the inability of the thin core to transfer heat away from itself, this section on some of the cores became hardened by sintering of the tungsten structure itself and by diffusion bonding with the columbium. Due to the inaccessability of the area for mechanical removal methods, tungsten removal from this area was particularly difficult.

Complete evaluation of the castings produced is described in subsequent sections of this report.

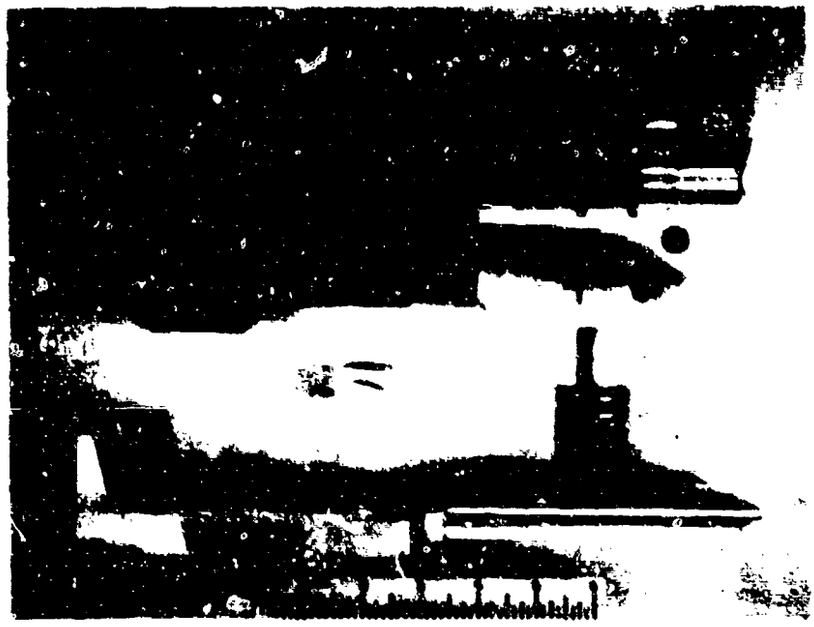


Figure 26. Wax Injection Tooling for Airfoil Configuration

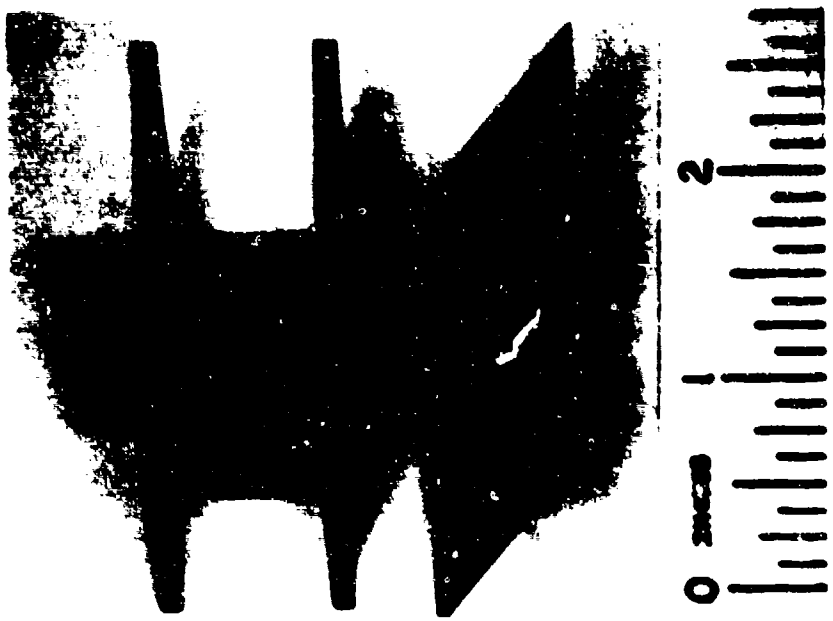


Figure 27a. Core Wax Pattern for Vane Having 0.040 Inch Wall

Figure 27b. Wax Patterns Having Solid Pattern (Left), 0.060 Inch Wall, and 0.040 Inch Wall (Right)

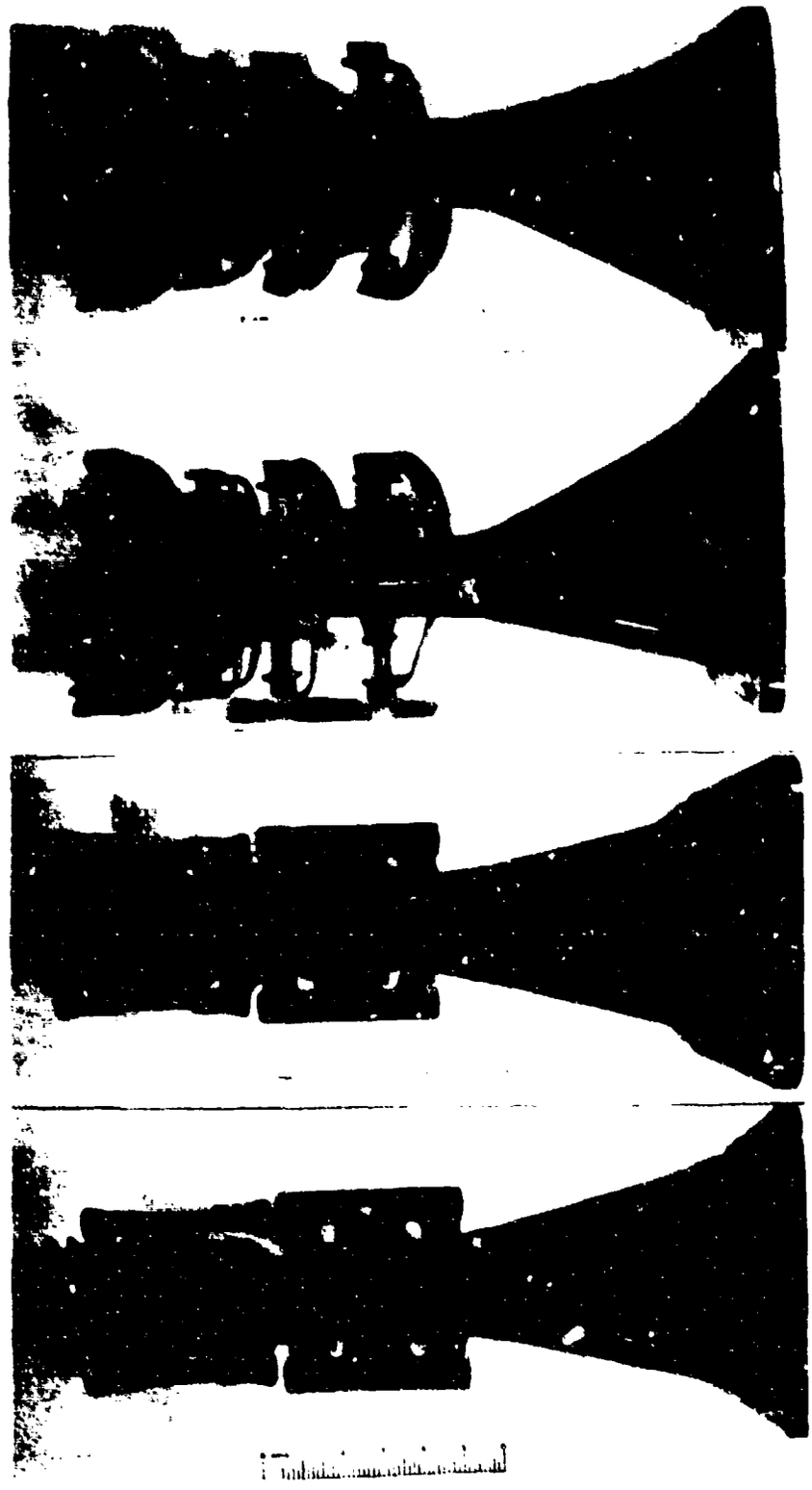


Figure 28. Initial Airfoil Mold Configuration

TABLE XI

FOUNDRY PARAMETERS FOR AIRFOIL CASTINGS

Parameters	Lot J	Lot K	Lot O	Lot P	Lot Q	Lot R
Crucible Size (dia)	6"	6"	6"	6"	6"	6"
Charge Weight	26.0 lbs	30.5 lbs	19.5 lbs	19.5 lbs	17.5 lbs	17.0 lbs
Vacuum At Start of Melt	15μ	25μ	20μ	10μ	10μ	20μ
Vacuum Rise During Melt	300μ	40μ	50μ	30μ	20μ	30μ
Furnace Hot Leak Rate	25μ/min.	17μ/min.	16μ/min.	19μ/min.	17μ/min.	17μ/min.
Final Skull Wt.	29.5 lbs	17 lbs.	17.5 lbs	16.5 lbs	16 lbs.	16 lbs.
Weight Melted from Electrode	23 lbs	23.5 lbs	37 lbs.	33 lbs	34 lbs	33 lbs
Weight Poured	19.5 lbs	36 lbs.	39.0 lbs	36 lbs	35.5 lbs	34 lbs
Power Input (Max. Amps)	8,000A	11,000A	12,000A	10,500A	10,000A	10,000A
Voltage (Max. Volts)	36V	40V	36V	41V	40V	38V
Power (Max. KW)	288	440	432	430	400	380
Melt Time	2 min., 38 sec.	1 min., 37 sec.	3 min., 12 sec.	2 min., 48 sec.	2 min., 48 sec.	2 min., 36 sec.
Time to Pour	3.1 sec.	2.9 sec.	3.1 sec.	3.4 sec.	3.4 sec.	3.4 sec.
Mold Temperature	1070°F	1940°F	1020°F	1005°F	1050°F	990°F

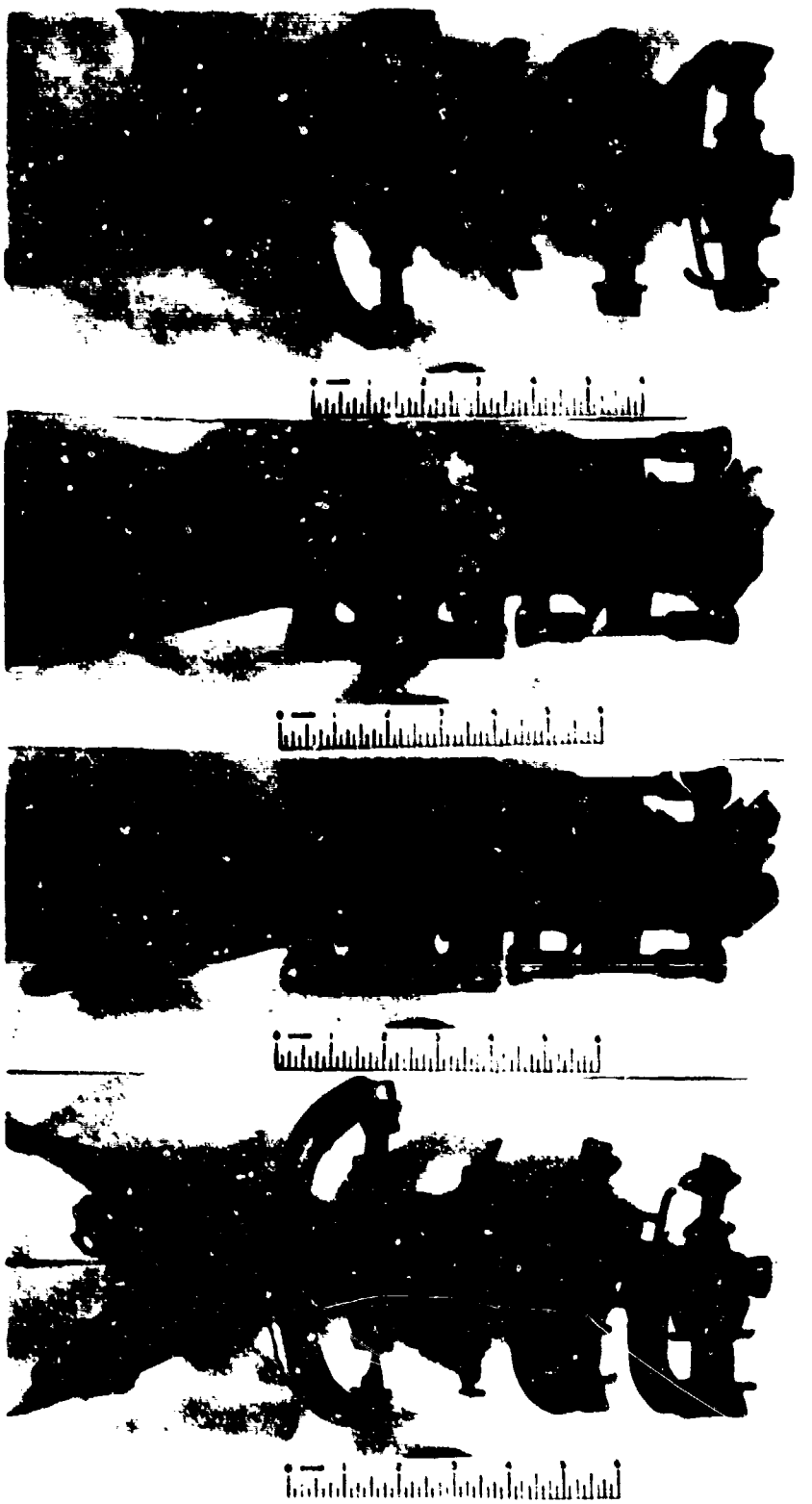


Figure 29. Lot J Casting After Partial Cleanup

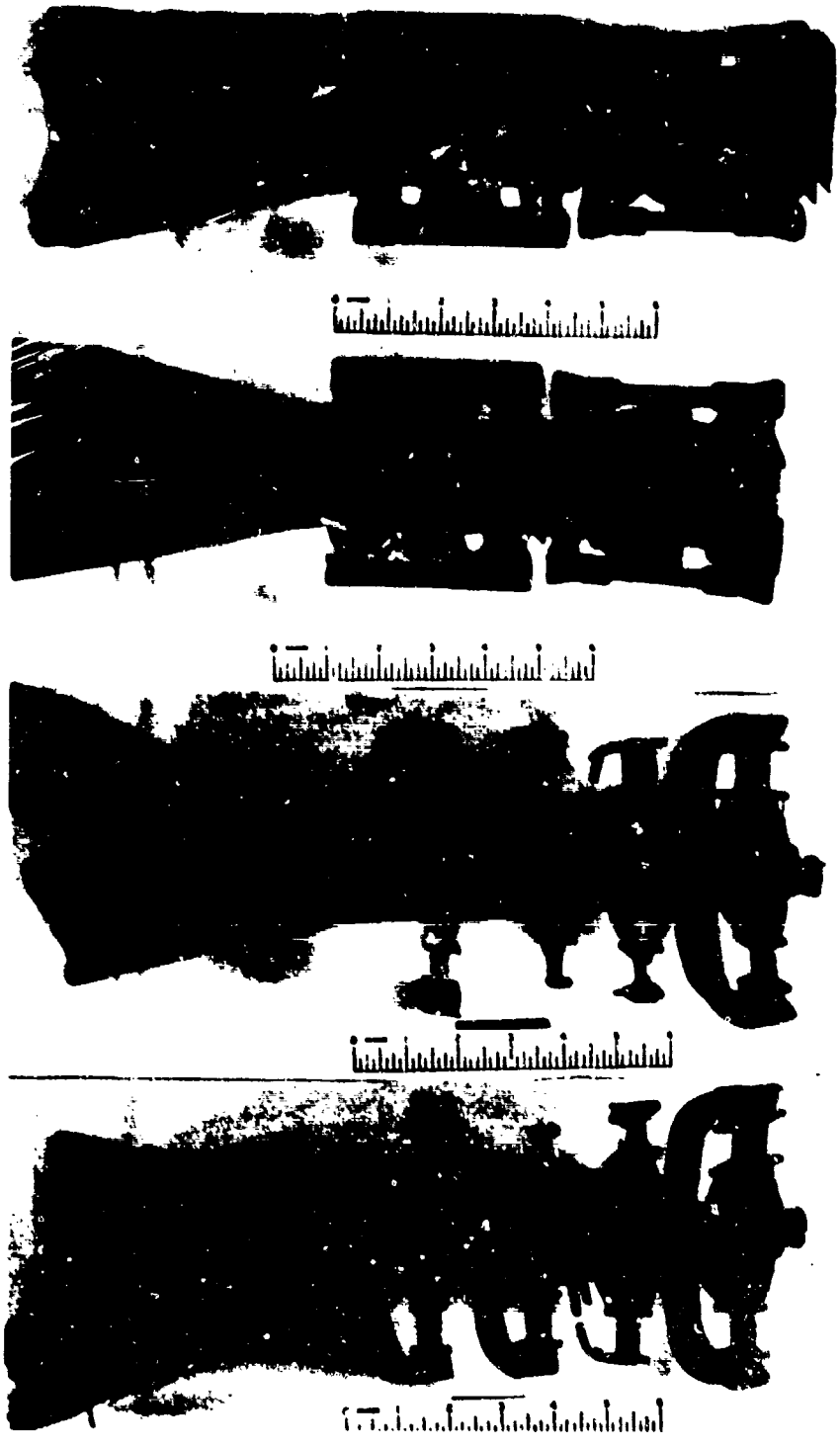


Figure 30. Lot K Casting After Partial Cleanup

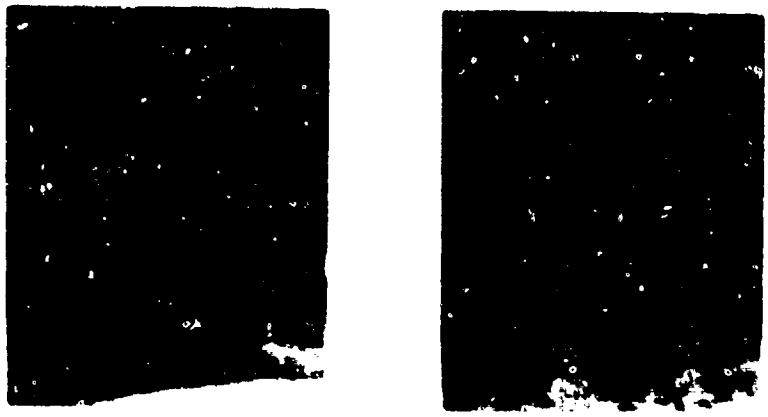


Figure 31a. Wax Pattern Assembly Lot O Mold

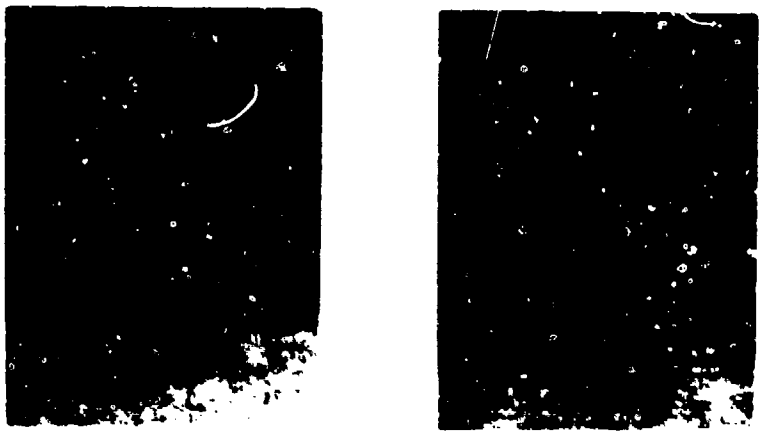


Figure 31b. Wax Pattern Assembly Lot P Mold

Figure 31c. Wax Pattern Assembly Lot Q Mold

Figure 31d. Wax Pattern Assembly Lot R Mold

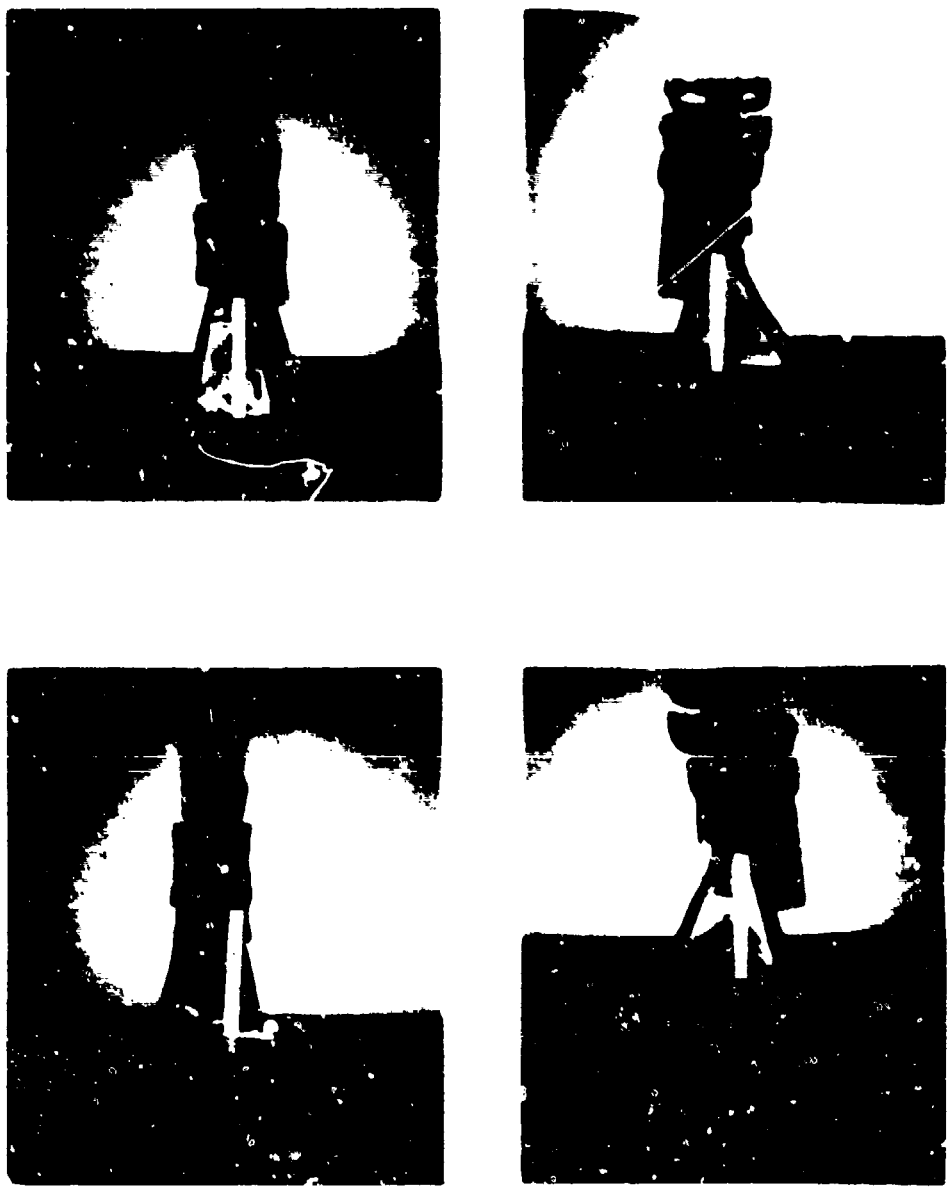


Figure 32a. Airfoil Castings Prior to Cutoff Lot 0 Castings

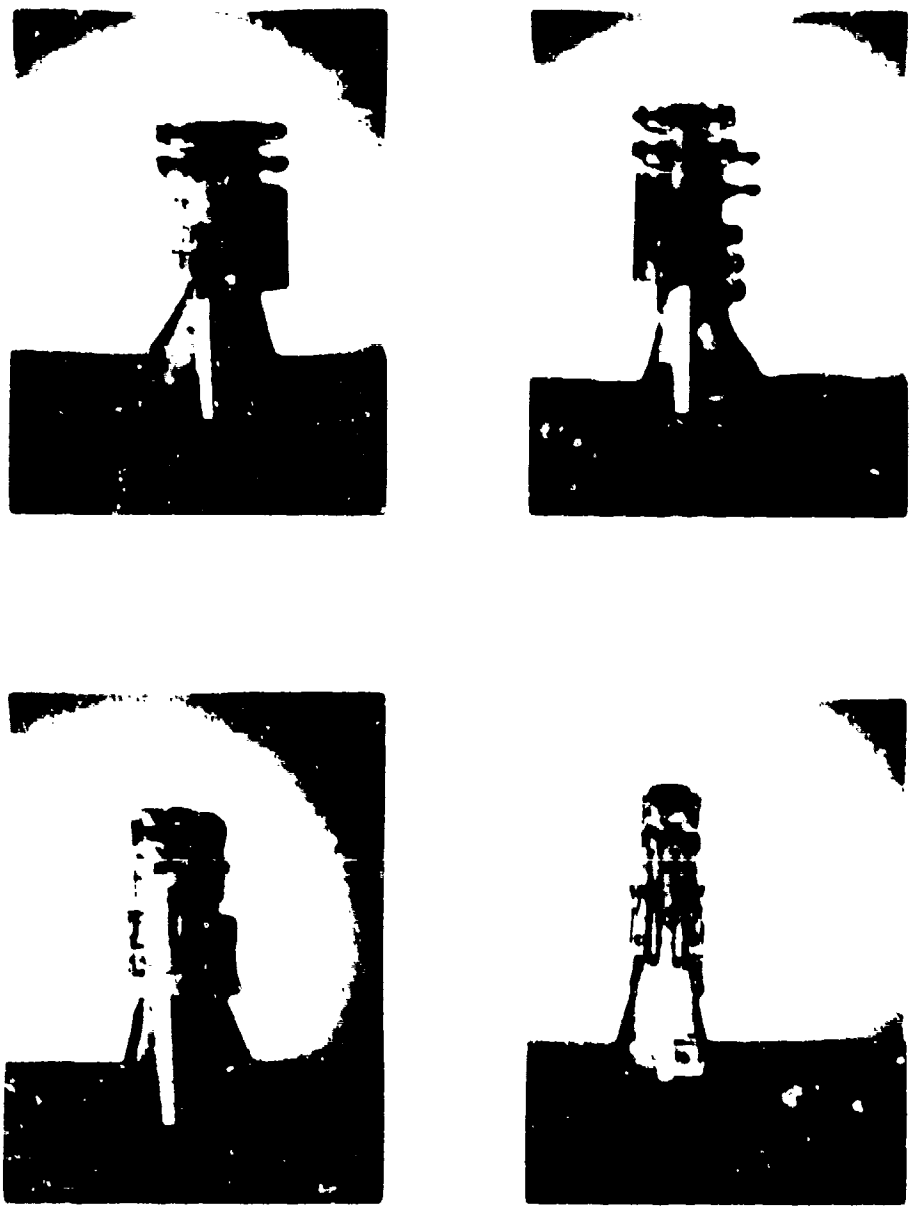


Figure 32b. Airfoil Castings Prior to Cutoff
Lot P Castings

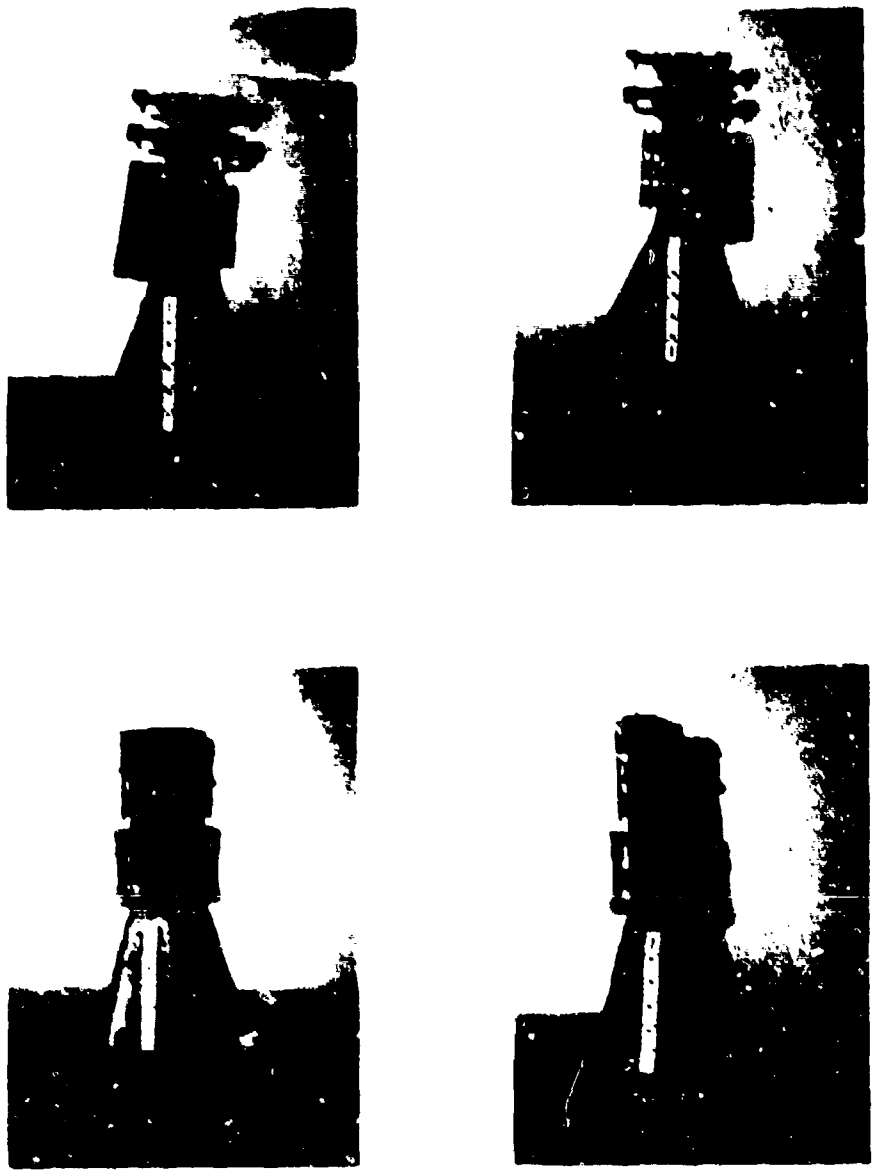


Figure 32c. Airfoil Castings Prior to Cutoff
Lot Q Castings

Figure 32d. Airfoil Castings Prior to Cutoff
Lot R Castings

SECTION X

NDT EXAMINATION

Throughout the course of the program, test specimen and airfoil castings were examined non-destructively to evaluate dimensions and types and quantities of discontinuities. One main objective in the initial castings was to determine if any of the alloys was different from the others with respect to quality of castings produced. Had there been a meaningful difference between alloys, this would have been a factor for consideration in alloy selection. Further evaluation of castings of C-103 provides correlation with mechanical properties, and characterize the effect of achievable defect levels on properties.

VISUAL INSPECTION

As shown in the photographs in previous sections, visual appearance of the casting was generally quite good. In most areas, the surface finish would be in the range 100-125 RMS, essentially the same as other types of production investment castings.

In the alloy comparison castings, discontinuities which did occur were essentially the same for each of the alloys. The most prevalent discontinuity was what is known as flow lines or cold lap. These originate due to premature freezing of the metal front during filling of the mold cavity. Ordinarily these are shallow enough that there is no functional degradation of the casting. In some cases, however, the defect is sufficiently deep so that surface blending or even weld repair of the area may be required to meet normal specification requirements. Such deep cold lap, cold shot, or cold shut usually would be identified and the approximate severity of the defect determined by fluorescent penetrant.

There were a few isolated areas of surface inclusions which would also potentially require repair in a finished casting.

The most outstanding problem identified by visual examination of the airfoil castings was a varying degree of incomplete fill in the thin walled airfoil sections. This, again, is a consequence of premature freezing of the metal, and is related to metal temperature, mold temperature, mold and gating configuration as well as the length-to-thickness ratio of the cavity being filled. Due to the melting method employed, meaningful variations in the metal temperature are not readily or controllably reproducible. Table XII summarizes the occurrence of no-fill in vanes of varying wall thickness, mold temperature and gating configureation.

In reviewing the degree of fill" data, it should be remembered that the wall thicknesses were chosen to determine the limit of producibility. The 0.060 inch wall was considered a reasonable goal, even though this is already quite thin. Reducing the wall thickness still further to 0.040 inch was not expected to result in complete

fill on any kind of a reliable basis, but was designed to represent the thinnest wall which could possibly fill completely based on existing technology.

In general, the solid vanes filled quite well, and the cored vanes with adequate gating also produced a complete part. With all other conditions the same, the 0.060 inch wall was more likely to fill than the 0.040 inch wall. While there is always some degree of probability involved in reproducibility of filling each configuration, the results generally fall in a pattern.

DIMENSIONAL INSPECTION

The major justification for specifying a precision casting is the ability of the process to produce a net or near-net component at a lower cost than by other methods. It is, therefore, of the utmost importance that dimensions of the casting meet the drawing requirements for production applications.

Dimensional measurements were made on test specimen and airfoil castings for purposes of characterizing shrinkage factors. Table XIII summarizes dimensions and calculated shrinkage factors for flat test specimens of each of the four alloys. The factors for the four alloys are considered in close enough agreement that the alloys may be considered essentially the same under these conditions. Table XIV summarizes measurements on selected C-103 airfoil castings.

FLUORESCENT PENETRANT INSPECTION

Test specimen and airfoil castings were inspected using Zyglo fluorescent penetrant. All of the defects identifed by fluorescent penetrant were identifiable visibly, as covered in the discussion of visual inspection. In most cases, visual irregularities did not give a penetrant indication, since the defects were sufficiently open.

Surface defects on the alloy comparison test specimen did not indicate that any of the alloys was better or worse for this characteristic. Penetrant or visual defects would have no effect on the actual test results included in this report due to the fact that all specimens were machined prior to testing.

Most surface discontinuities which were identified by penetrant inspection of the vanes were relatively shallow and minor surface blending was sufficient to remove the defect without a gross change in casting dimensions. Some isolated areas were sufficiently severe that weld repair was required.

RADIOGRAPHIC INSPECTION

X-ray parameters were established for each of the alloys after

several experimental iterations. Owing particularly to the varying tungsten content of the alloys, sensitivity and penetration capability had to be determined and parameters adjusted for each alloy.

The results of x-ray examination of the alloy comparison test specimens are reported in Table XV. Both the flat and round specimen molds had been prepared with an absolute minimum of gating. This was intended not to produce sound castings of all alloys, but to determine relative feeding characteristics of the four alloys. In reviewing the results, it it concluded that the four alloys are essentially equivalent in directional solidification and feeding characteristics.

Test specimens cast subsequent to selection of C-103 were also radiographically inspected. For these specimens, the gating was changed to reflect best practice for producing a sound casting. These specimens in general were sound, particularly the round specimens. Shown in Appendix 2 are the tensile properties and x-ray characterization of each of the test bars which was tested. In this case, the x-ray was taken after machining the bar and reflects the condition in which it was tested. Tensile specimens tested in the latter half of the program were essentially free of internal defects.

Those vane castings which were completely filled out were x-rayed and evaluated for internal defects. The solid airfoils were quite sound and those having the internal core had varying degrees of internal porosity. Within the airfoil section itself, many of the castings met ordinary aerospace requirements for soundness. Gating design was concentrated mainly on filling the castings, with internal soundness being a secondary goal. Radiographs showing the fillet radius between the airfoil and platforms show a relatively large amount of shrinkage in this "T" section in the cored vanes. This is a consequence primarily of the chilling effect of the core. Elimination of shrinkage in this area of the cored castings would require additional gating optimization and possibly minor design change, which would best be done in the context of producing a component designed for actual engine use.

METALLOGRAPHIC EXAMINATION

While not strictly part of non-destructive evaluation, photomacrographs of the vanes are included in this section for reference purposes. See Figure 33. One airfoil of each configuration was sectioned at approximately the mid-point of the airfoil, metallographically prepared and etched to show the grain structure of the casting. The etching solution used was the same as previously used for photomicrographic preparation (Section V). In each case, a very fine grain size is observed. The average grain diameter measured through the wall thickness is shown in Table XV.

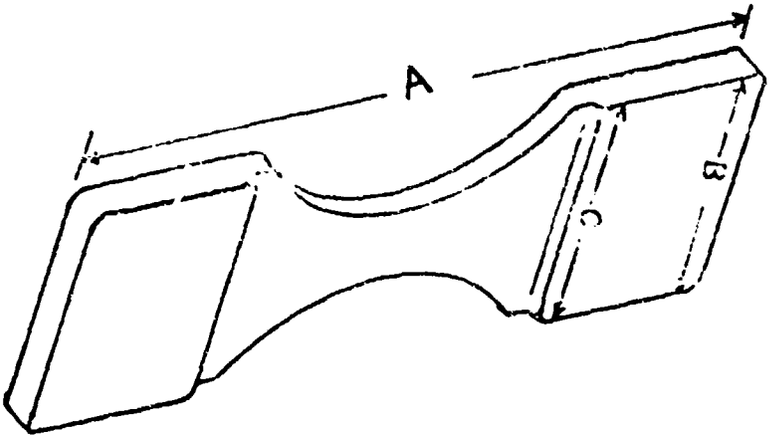
TABLE XII

CORRELATION OF AIRFOIL GATING
CONFIGURATION AND DEGREE OF FILL

Mold Temp. °F	Cored Or Solid	Wall Thickness	Gating Configuration	Airfoil No-Fill Description
1020 to 1070°F	Solid	---	Large Gate at one platform only and wax drain at the other end.	Gross no-fill at the trailing edge.
1020°F 1070°F	Solid	---	Large gates on both ends.	Minor no-fill at the trailing edge.
1940°F	Solid	---	Large gate at one end only and wax drain at the other end.	Minor no-fill at the trailing edge.
1940°F	Solid	---	Large gates on both ends.	Airfoil section completely filled.
1005 to 1070°F	Cored	0.060"	Two large gates at one end only and wax drain at the other end.	Gross no-fill of the platform and airfoil on the specimens that are in the bottom in the casting positions. The airfoils with extra thickness built in at the trailing edged filled out good.
1005 to 1070°F	Cored	0.060"	Two gates on one platform and three on the other.	Major no-fill of the airfoils.
1940°F	Cored	0.060"	Two gates at one end and wax drain at the other end.	Minor no-fill.
1940°F	Cored	0.060"	Two gates on one plateform and three on the other.	Airfoils filled out well.
1050°F to 1070°F	Cored	0.040"	Two gates at one end only and wax drain at the other.	Gross no-fill of airfoil and platform.
1050°F to 1070°F	Cored	0.040"	Two gates at one end and three at the other with the airfoil being at the top of the mold in the as-cast position.	Major no-fill of the airfoil.
1050°F to 1070°F	Cored	0.040"	Same gating as above; but the airfoil at the bottom of the mold.	Generally good fill out - with some very minor holes in the airfoil.
1940°F	Cored	0.040"	Two gates at one end and wax drain at the other.	Major no-fill.
1940°F	Cored	0.040"	Two gates at one end and three at the other end.	Some minor no-fill of the airfoil.

TABLE XIII

DIMENSIONAL INSPECTION OF FLAT TEST SPECIMEN BLANKS



Actual Dimensions, Inches

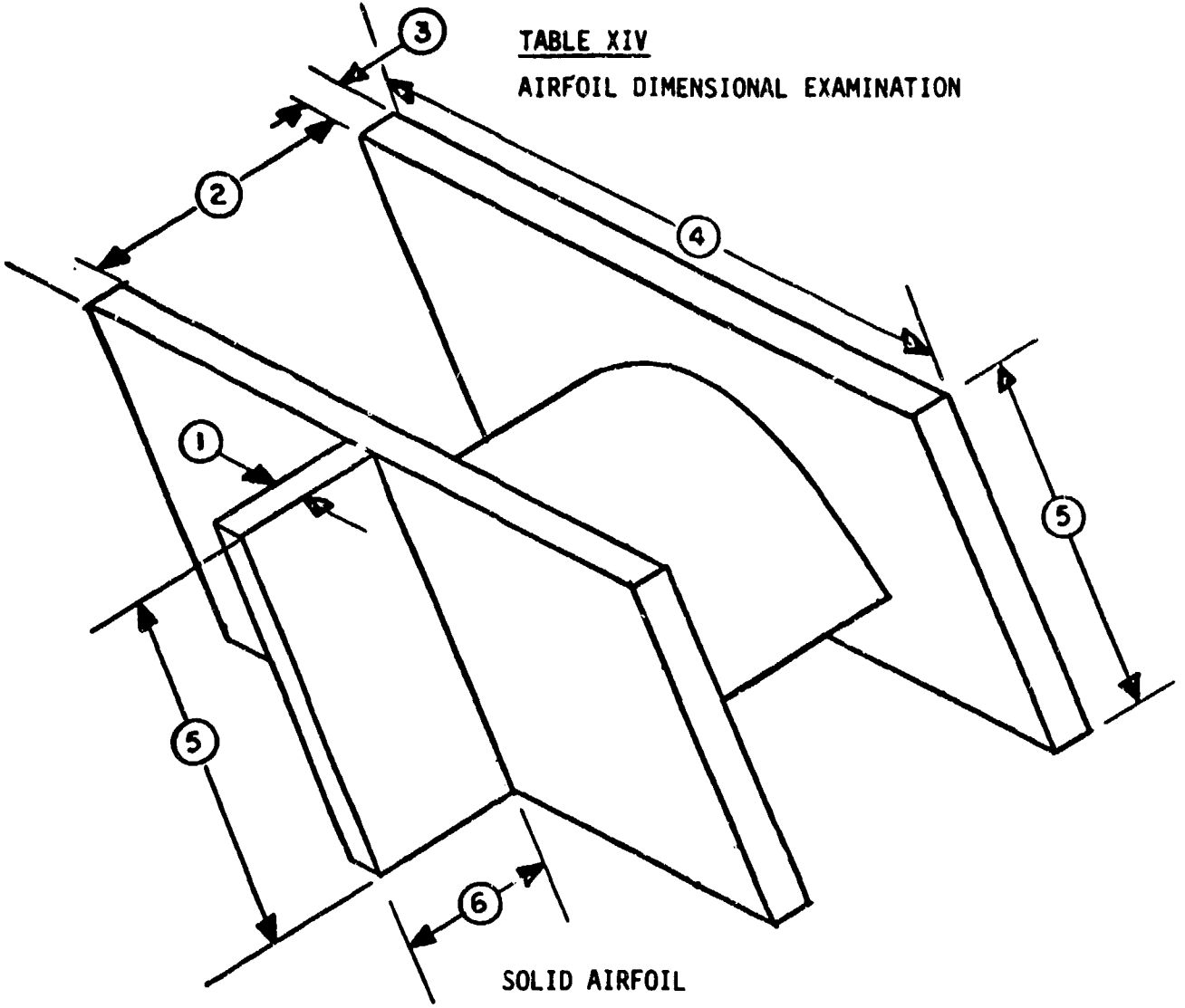
Alloy	Tool			Wax			Casting		
	A	B	C	A	B	C	A	B	C
C-103	3.630	1.215	1.213	3.611	1.203	1.200	3.563	1.194	1.191
Cb-752	3.630	1.215	1.213	3.610	1.204	1.200	3.544	1.191	1.188
C-129Y	3.630	1.215	1.213	3.609	1.202	1.200	3.562	1.190	1.189
SU-31	3.630	1.215	1.213	3.611	1.204	1.200	3.563	1.197	1.193

Calculated Shrinkage Factor, Inches Per Inch

Alloy	Shrink From Wax to Cast		Shrink From Tool to Cast		Composite Shrinkage Factor
	Long. Per In.	Trans. Per In.	Long. Per In.	Trans. Per In.	
C-103	0.013	0.007	0.018	0.017	.014
Cb-752	0.015	0.010	0.021	0.020	.016
C-129Y	0.013	0.009	0.020	0.019	.015
SU-31	0.013	0.006	0.018	0.016	.013

TABLE XIV

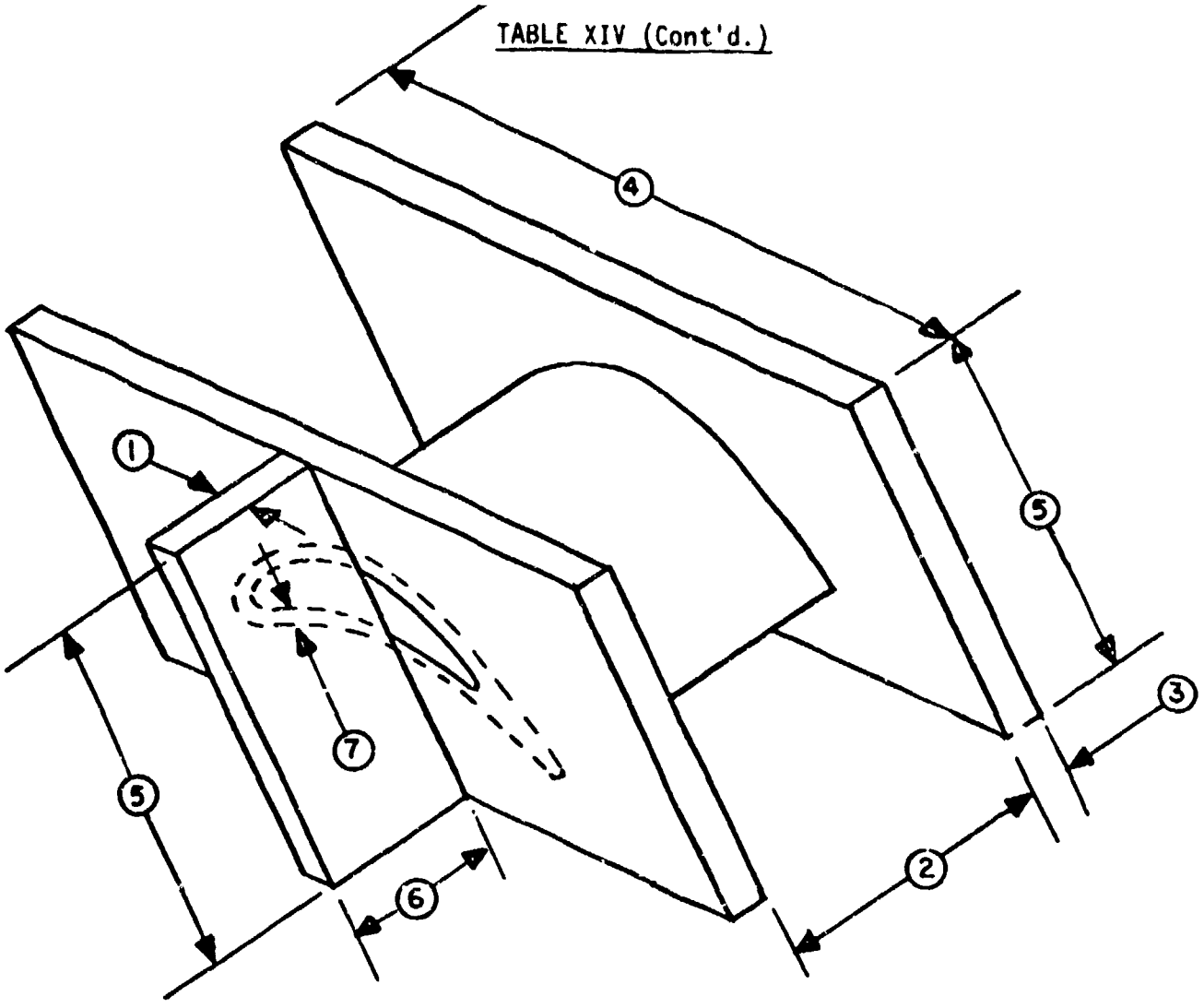
AIRFOIL DIMENSIONAL EXAMINATION



SOLID AIRFOIL

Dimension	Tool	Wax	Casting	Shrinkage Factor		
	Inch	Inch	Inch	Tooling to Casting in/in	Wax to Casting in/in	Composite in/in
1	.091	.091	.089	.022	.022	
2	.820	.816	.807	.016	.011	
3	.113	.112	.109	.035	.027	.024
4	2.127	2.120	2.060	.031	.028	
5	1.064	1.059	1.040	.023	.018	
6	.460	.457	.450	.022	.015	
Average				.025	.020	

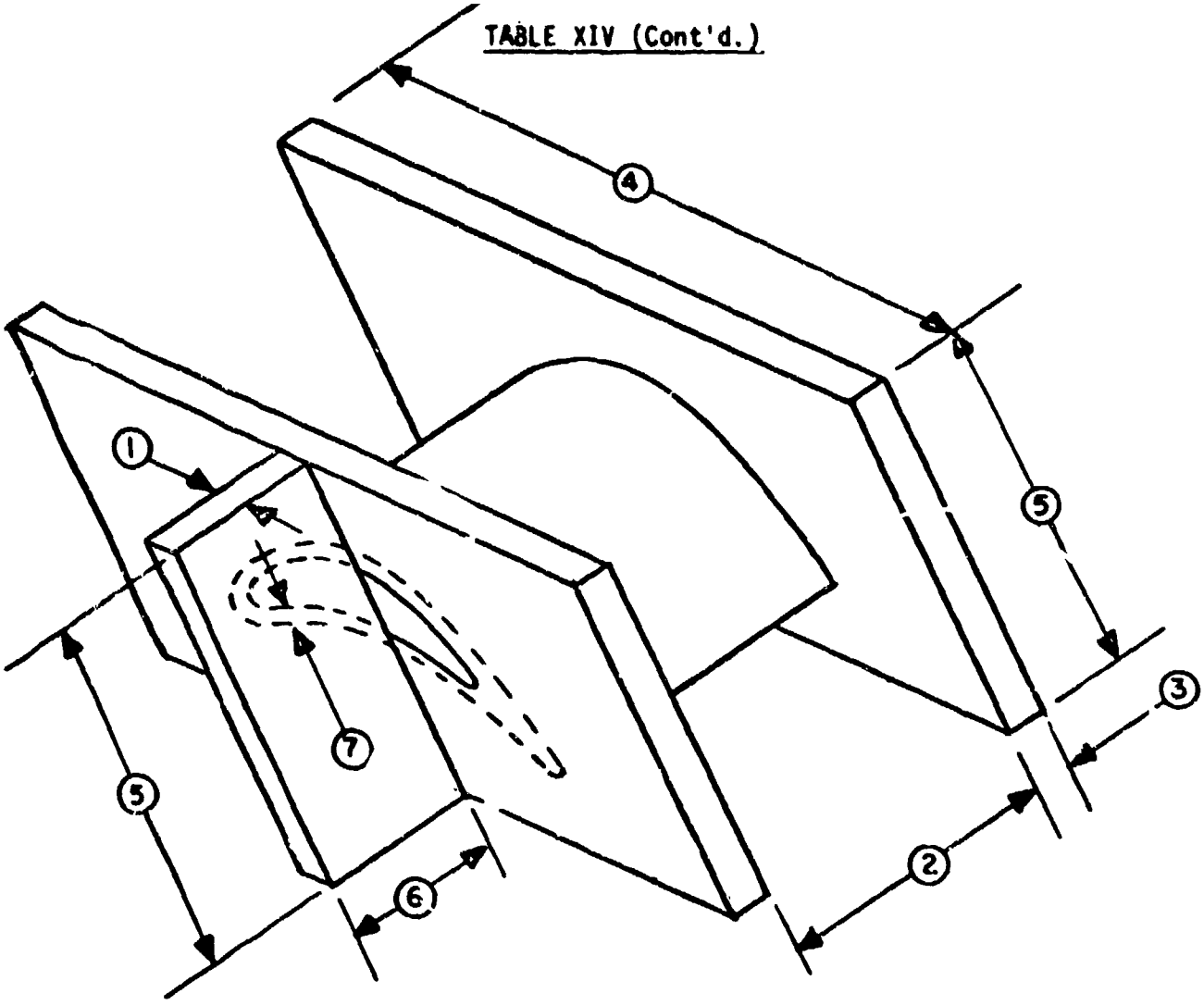
TABLE XIV (Cont'd.)



AIRFOIL WITH 0.060" WALL

Dimension	Tool	Wax	Casting	Shrinkage Factor		
				Tooling to Casting in/in	Wax to Casting in/in	Composite in/in
	Inch	Inch	Inch			
1	.091	.091	.089	.022	.022	
2	.820	.816	.806	.017	.012	
3	.113	.112	.110	.027	.018	
4	2.127	2.120	2.061	.031	.028	024
5	1.064	1.059	1.039	.023	.019	
6	.460	.457	.450	.022	.015	
7	.059	.058	.057	.034	.017	
Average				.025	.019	

TABLE XIV (Cont'd.)



AIRFOIL WITH 0.040" WALL

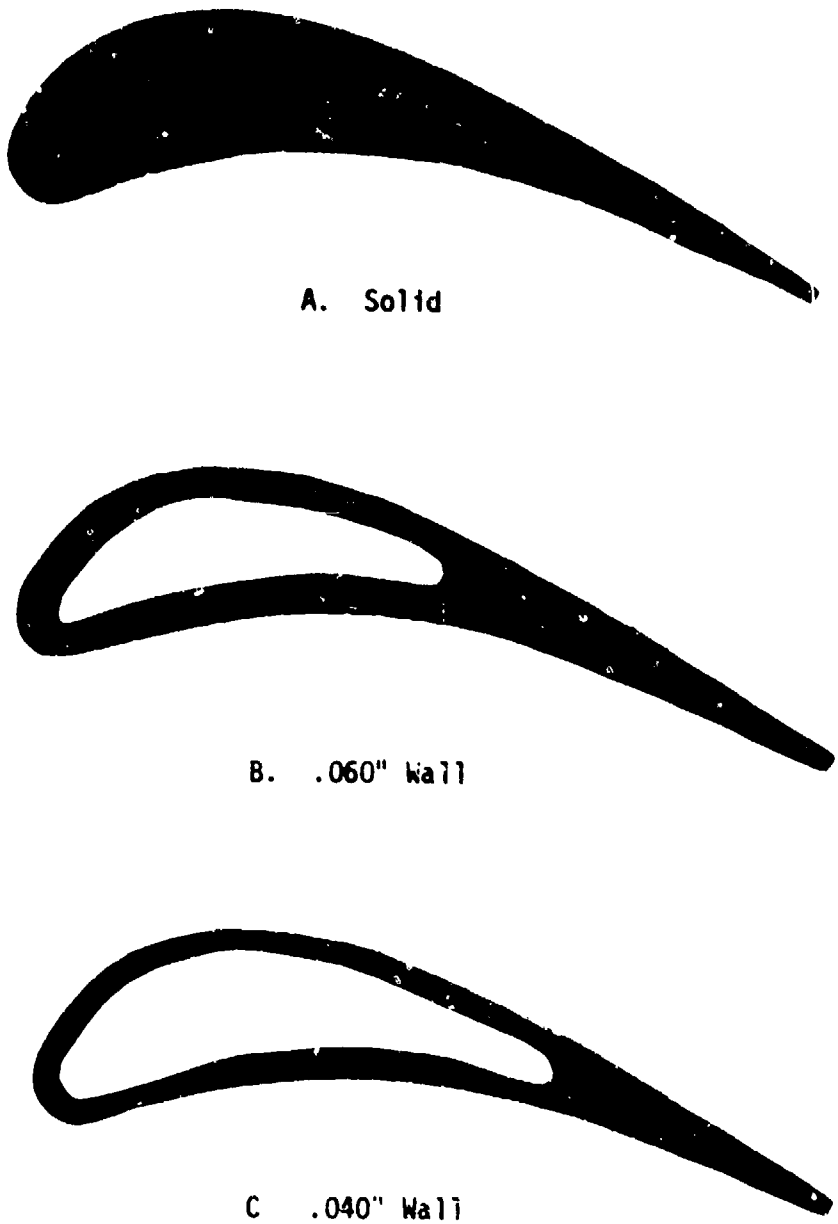
Dimension	Tool	Wax	Casting	Shrinkage Factor		
				Tooling to Casting in/in	Wax to Casting in/in	Composite in/in
	Inch	Inch	Inch			
1	.091	.091	.089	.022	.022	
2	.820	.816	.806	.017	.012	
3	.113	.112	.110	.027	.018	
4	2.127	1.122	2.062	.030	.028	.024
5	1.064	1.059	1.040	.023	.018	
6	.460	.457	.449	.024	.018	
7	.037	.037	.036	.027	.027	
Average				.024	.020	

TABLE XV

RADIOGRAPHIC COMPARISON OF ALLOY FEEDING CHARACTERISTICS
ASTM E-155 Plate Numbers Corresponding to the
Maximum Defect Size in the Radiographs

S/N	Size	Alloy	Gas 1·1	Shrink 2·1	Foreign Material 3·12
1	½" round	SU-31	2 (None)	1	None
2	½" round	SU-31	None	2	None
3	½" round	SU-31	1	2	None
4	½" round	SU-31	1	1	None
5	½" round	SU-31	1	1	None
6	½" round	SU-31	2 (1)	2	None
9	5/8" round	SU-31	1	1	None
10	5/8" round	SU-31	6	1	None
3	.090" flat	SU-31	1	3	None
4	.090" flat	SU-31	None	4	None
7	½" round	Cb-752	1	1	None
8	½" round	Cb-752	1	None	None
9	½" round	Cb-752	1 (1)	1	None
10	½" round	Cb-752	7	2	None
11	½" round	Cb-752	1 (None)	1 (1)	None
12	½" round	Cb-752	1	1	None
11	5/8" round	Cb-752	1	1	None
12	5/8" round	Cb-752	None	1	1
5	.090" flat	Cb-752	1	3	None
6	.090" flat	Cb-752	1	3	None
13	½" round	C-103	1 (None)	None	None
14	½" round	C-103	3	1	None
15	½" round	C-103	None	2	None
16	½" round	C-103	2	1	None
17	½" round	C-103	1 (1)	3	None
18	½" round	C-103	None	1	None
13	5/8" round	C-103	None	1	None
14	5/8" round	C-103	None	None	None
7	.090" flat	C-103	3	4	None
8	.090" flat	C-103	2	3	None
19	½" round	C-129Y	1	2	None
20	½" round	C-129Y	6	2	None
21	½" round	C-129Y	1	2 (2)	None
22	½" round	C-129Y	None	1	None
23	½" round	C-129Y	None	2	None
24	½" round	C-129Y	None	1 (1)	None
15	5/8" round	C-129Y	6	1	None
16	5/8" round	C-129Y	None	1 (None)	None
9	.090" flat	C-129Y	1	4	None
10	.090" flat	C-129Y	1	4	None

Numbers in the parentheses refer to the defect sizes present in the test bars after machining to ⅛" size as Per ASTM E8-69 for tensile tests.



A. Solid

B. .060" Wall

C .040" Wall

3.3X

Figure 33. Macrostructure of C-103 Airfoil Castings

TABLE XVI

GRAIN SIZES FOR VARIOUS TYPES OF AIRFOILS

Airfoil Configuration	Average Grain Diameter	ASTM Grain Size
Solid	.010"	1
.060" Wall	.006"	2½
.040" Wall	.005"	3

SECTION XI

HEAT TREAT OPTIMIZATION

A detailed study of the effect of heat treatment on microstructure and hardness was reported in Section V. Based upon those findings, an additional investigation was carried out in the areas of heat treat response, cooling rate effects, and post weld heat treatment.

HEAT TREAT RESPONSE

As shown in the metallographic study, C-103 did show a relatively good measure of response to solution treating in the range 2550°F to 2900°F and to aging in the area of 2200°F. This was seen in the micrographs and to some extent in the hardness measurements. While an extensive mechanical property versus heat treatment study of all the alloys was beyond the scope of the program, some additional tests were run for C-103.

The heat treatment schedule selected for the mechanical property tests was slightly different from the conditions used for the metallographic study. Selection of that cycle was based both upon the results of that study and on consideration of subsequent processing of the castings prior to actual use. While heating and cooling rates were not necessarily the same, the temperature and time cycles were chosen to achieve the desired microstructural changes and actually be consistent with the time and temperature cycles used in applying one of the more promising oxidation resistant coatings. This can be considered in two ways. Firstly, the behavior of a non-heat treated casting during the coating cycle is elucidated, and secondly, with appropriate behavior of the material, the coating cycle can be considered to serve a double purpose (i.e. coating and heat treating).

The specific heat treatment parameters selected were as follows:

2760°F for one hour, furnace cool, plus
2100°F for three hours, furnace cool

Appendix 2 includes a direct comparison of as-cast and heat treated properties for specimens cast along with the airfoil castings. The low ductility in the non-heat treated castings will be discussed later in this section.

Based upon these tests, the time-temperature schedule, whether used as heat treatment alone or presumably as the coating cycle, appears advantageous in maximizing the mechanical properties of the alloy.

The following is a tentative explanation of what occurs during the solution and age cycle, and why it is beneficial to mechanical

properties. To a first approximation, the behavior of C-103 during the heat treatment cycles used is very similar to that in steels. Upon heating to a solutioning temperature, the precipitate goes into solution, resulting in a relatively clean microstructure with relatively few precipitates remaining. Upon "quenching" or, cooling rapidly enough from the solution temperature, retention of the oxides, carbides, etc. in solution creates a supersaturated condition with relatively high internal stresses, poor ductility, and elevated hardness resulting. Upon aging, or tempering, the material is heated to a temperature at which precipitation can occur at a reasonable rate, and precipitation occurs. In the case of the observed C-103 behavior, as with steels, tempering, or aging forming the precipitates, actually produces a softer and more ductile material. Generally, the strength would also be expected to go down, which appears to happen to some extent.

Along with simply solutioning and aging, another phenomenon occurs which makes the heat treatment even more beneficial. In the as-cast structure, the precipitation which has occurred is primarily at or near the grain boundaries which is the most detrimental area with respect to effect on ductility. Within the grains, there are sub-grains having slightly different orientation than other areas of the same grain. This produces a network of sub-grain boundaries which are potential precipitation sites, although in the as-cast structure they are relatively free of precipitate. Figure 36 shows fairly clearly some of these sub-boundaries having precipitate in an as-cast structure, but this is a typical. During the solution and age cycle the precipitate is dissolved and then allowed to reprecipitate at the most available grain boundary or sub-boundary, dispersing it more than it was in the as-cast material. A simple comparison of Figure 34 (heat treated) with Figures 35 or 36 (as-cast) leads to the impression that the heat treatment reduces the grain size when in fact the sub-boundaries shown in Figure 34 are actually present as-cast but are just very difficult to see due to lack of precipitation at these sub-boundaries. The same etching solution composition was used for preparation of these samples as was used for previous samples (See Section V).

(Note: Figures 34, 35, and 36 were taken from the shoulder of the test bars marked with an asterisk in Appendix 2.)

COOLING RATE EFFECTS

One phenomenon which was somewhat puzzling was the variation in ductility of C-103 castings which was only partially explainable in terms of section thickness effects. Pre-program data on C-103 indicated a relatively high ductility of >20% elongation with good reliability. Some of the program results were much lower, and a satisfactory explanation was not forthcoming until the relationship of microstructure and properties was understood. Of the test bars cast during the latter half of the program, none of the as-cast

specimens resulted in elongation greater than 20 percent. Indeed, most were yielding values in the range of 5 - 10 percent.

In an effort to determine the effect of mold temperature, one of the airfoil molds had been well insulated and heated to nearly 2000°F, compared to the normal nominal temperature of 1000°F. Importantly, this resulted in a much slower cooling rate than experienced by the remainder of the airfoil molds, with the resulting microstructure comparison in Figures 35 and 36. It should be noted, that in Figure 36 a greater degree of aging occurred during slower cooling through the precipitation range, and a substantially higher ductility was attained. Previous observations of ductility and casting conditions are in reasonable agreement with this explanation.

WELDABILITY AND POST-WELD HEAT TREATMENT

Consideration of weldability is an exceedingly important factor in anticipating production useage of columbium alloy castings. In many current and projected applications, columbium components, whether castings or machined parts, are joined by electron beam or tungsten inert gas welding into a final assembly. In addition, due to the high relative cost of a columbium casting, the justification for repairing otherwise unuseable castings is very easy to see, as long as it is performed in a technically correct and adequately controlled manner. The reluctance of some users to accept weld repair is usually based upon a experience with materials which are either unsuitable for weld repair or which were weld repaired in an uncorrect manner. The overall generalization that weld repair is not good is often not based upon sound judgment of specific technical factors nor on economic realities. In the case of columbium alloy castings, judicious use of weld repair techniques will be a prerequisite for full realization of the cost savings to be achieved by use of the casting process.

Pre-program tests had indicated that weld repair or weld joining of C-103 castings or assemblies would produce a material of strength equivalent to as-cast with some reduction in ductility. After establishing an understanding of the influence of heat treat condition on C-103 microstructure and properties, this background was applied to the situation of a component or an assembly containing a weld. As discussed above, rapid cooling from melting temperatures results in a relatively hard, precipitate-free microstructure having reduced ductility. In the case of a weld, a most severe cooling rate is experienced by the weld material. The surrounding "heat affected zone" also is heated to some elevated temperature and cooled quickly. The effect of the difference between weld and as-cast conditions is magnified when both conditions are found in the same test specimen. Since material in the two conditions is not of the same strength, one area will yield before the other reaches its yield strength. It was anticipated that application of the successful heat treatment

cycle to a welded structure would "homogenize" the total specimen so that properties equivalent to "as-cast plus heat treat" should be obtained.

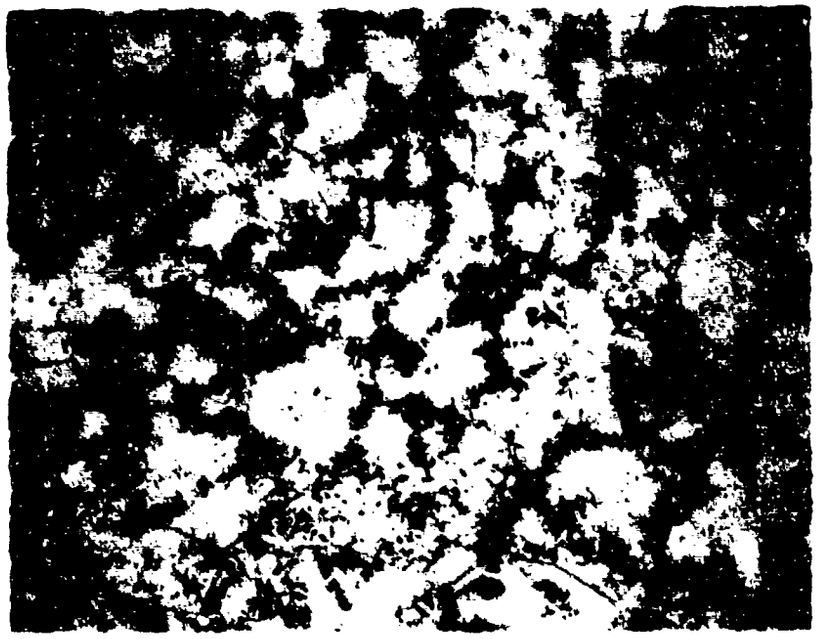
Tensile specimens of C-103 having no detectable internal discontinuities were ground in the same manner as a casting would be ground to remove a defect prior to weld repair. Fully one-half of the cross section of the test bar was removed at the center of the gage section, using a round carbide grinder. A photo of a typical weldability test specimen prior to welding is shown in Appendix 3.

After appropriate cleaning, each specimen was weld repaired using the tungsten inert gas technique in a dry box which was backfilled with high purity argon. Filler metal was used which had the same nominal composition as the casting. Differences in exact composition both of alloying elements and interstitials would have a second order effect on properties, but these variations were not considered in the current tests.

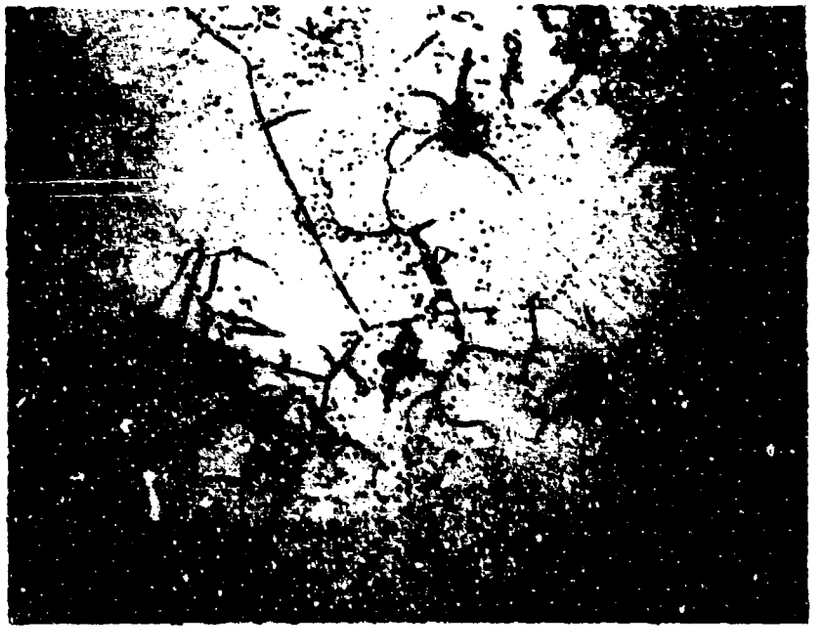
The weld repaired specimens were machined to final test dimensions, either with or without heat treatment, and were tested at room temperature or 2200°F. The results of these tests are shown in Appendix 2. It can be seen that the as-cast plus welded specimen continues to meet the program minimum of 5 percent elongation. The heat treated specimen showed a great improvement in ductility, with a corresponding reduction in strength. Corresponding to the increase in ductility, the mode of fracture was also changed by application of the post-weld heat treatment.

The fracture surfaces of the room temperature specimens are shown in Figure 37. In the as-welded specimen, the fracture was largely intergranular. The columnar grains of the weld, in which fracture occurred, can be readily seen. The heat treated specimen fractured in approximately the same location, but was predominately transgranular. This is hypothesized to be a consequence of redistribution of the precipitates away from the grain boundaries.

In general, the test results indicate that the thermal history of C-103, including casting cooling rate, weld repair, overheating during cut off, etc., may have a detrimental effect on properties of the casting. Heat treatment through a solutionizing and subsequently an aging cycle restores and even improves upon the as-cast properties. While other time and temperature combinations may ultimately prove to be optimum, the conditions used in this work have proven to be very effective.



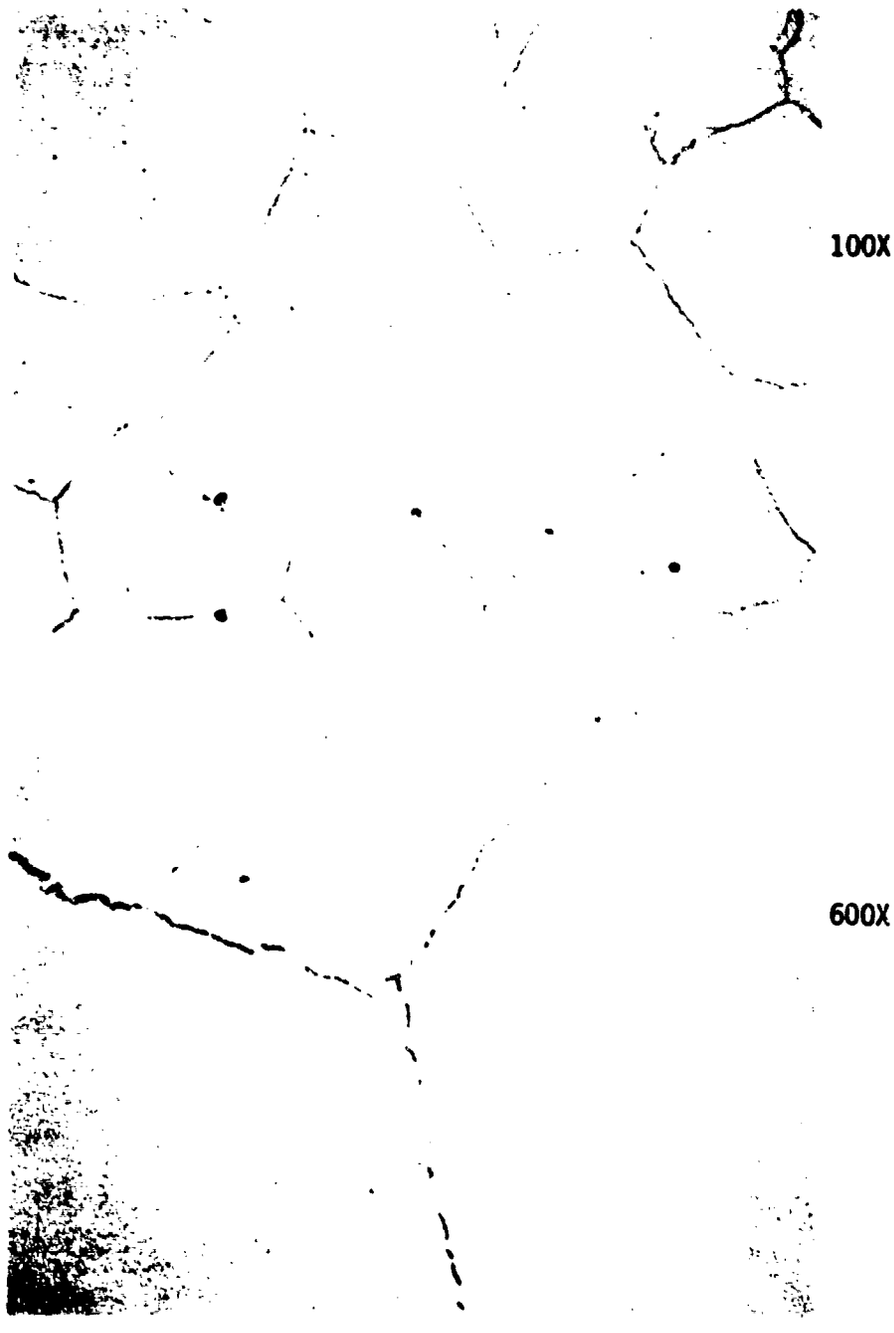
100X



600X

Average R.T. Tensile Properties 58.8, 39.5, 26.3, 39.3

Figure 34. C-103 Heat Treated Microstructure
2760°F, 1 Hr., Furnace Cool plus
2100°F, 3 Hrs., Furnace Cool



Averaged R.T. Tensile Properties 59.0, 44.6, 7.1, 7.2

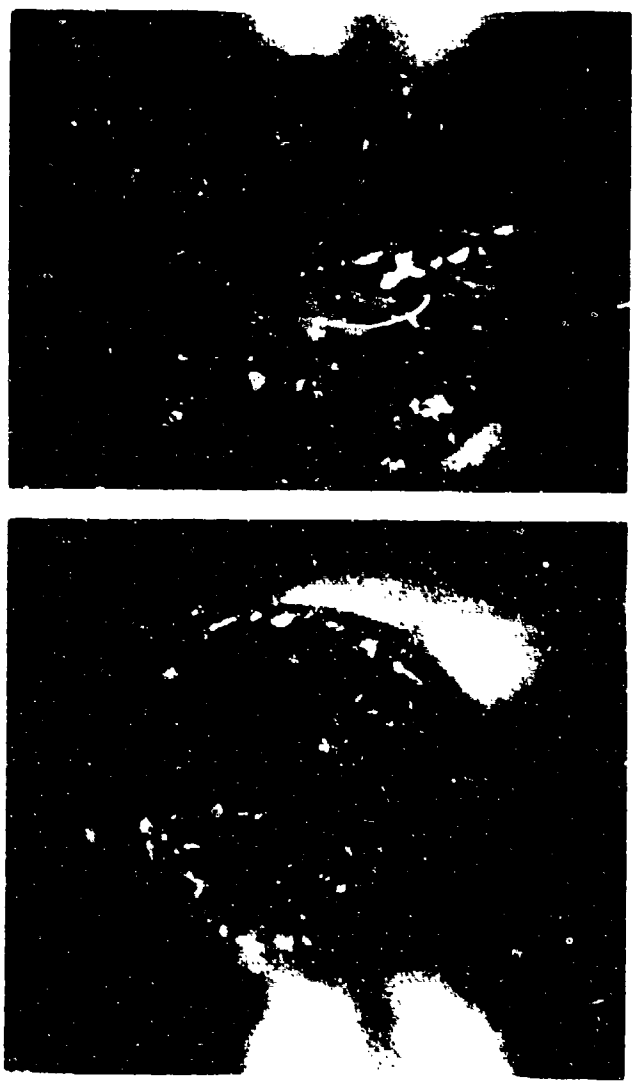
Figure 35. C-103 As-Cast Microstructure Rapid Cooled

100X

600X

Average R.T. Tensile Properties 62.1, 44.2, 16.5, 20.0

Figure 36. C-103 As-Cast Microstructure Slow Cooled



AS WELDED

10X

(Reduction in area 10%)

HEAT TREATED AFTER WELDING

10X

(Reduction in area 54%)

Figure 37. Fracture Surfaces of Welded Test Specimens

SECTION XII

CONCLUSIONS AND RECOMMENDATIONS

The following paragraphs summarize the conclusions which are drawn from program results and recommendations based upon these conclusions:

1. With the exception of a melt-initiation problem with C-129Y, the four alloys, C-103, SU-31, Cb-752, and C-129Y are very similar with respect to castability, fluidity, metal flow, and feeding characteristics. This may be further extended to a statement that most columbium alloys, with some exceptions, will be reasonably alike in these characteristics.

2. Chemical composition of the columbium alloys is not changed in an unpredictable or excessive manner. Alloying elements show virtually no change as a result of melting and casting. Interstitial elements, particularly oxygen, show some increase.

3. Direct recycling of foundry scrap by the foundry will be required in order to make columbium castings cost competitive with other production methods. Test results demonstrate the feasibility of indefinite recycle with addition of new melt stock to replace material in castings shipped.

4. Mechanical properties of the columbium alloys in the as-cast form are generally lower than the typical values for wrought material. Two of the four alloys demonstrated capability of meeting program mechanical property goals both at room temperature and at 2200^{0}F without any heat treatment.

5. Response of the four alloys to heat treatment was investigated and different results were obtained for each. More detailed investigation of C-103 revealed a very good response in mechanical properties versus heat treatment, allowing projection of the ability to expect tensile property minimums the same as those for bar stock even for weld-repaired or weld-joined castings. For applications where a higher strength alloy is required, further heat treatment studies of the other alloys and particularly SU-31 would undoubtedly prove rewarding.

6. Airfoil castings as shown in Figure 38 can be produced with an internal core and having a wall thickness as low as 0.040 inches, although at any given time there will be a trade off between a thinner wall and a higher casting quality and/or yield. Having adequately demonstrated the feasibility of producing such castings, it is recommended that airfoil castings be produced having a practical configuration which can be coated and thoroughly tested in an actual engine environment.

7. A tentative specification has been established for C-103 castings. This specification will certainly require changes and up-dating prior to use for procurement of castings on a production basis. However, it does provide a basis against which specific components and applications may be considered, and from which improvements may be made.

This program has provided a major step forward in the technology of columbium alloy investment casting, and equally important in the characterization of the castings produced.

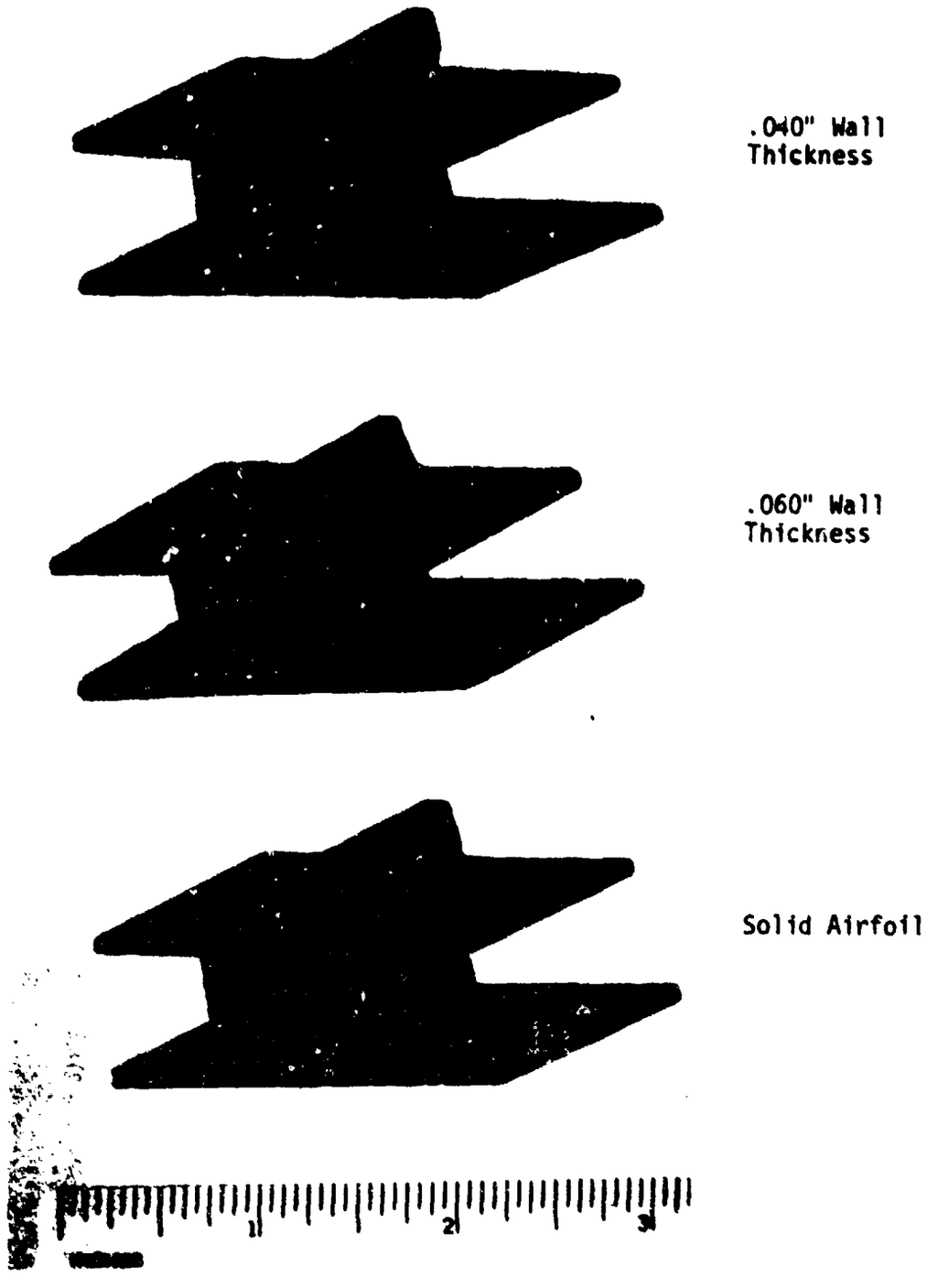


Figure 38. Investment Cast Columbium C-103 Turbine Vanes

APPENDIX 1A
Chemical Analysis Tabulation
Columbium Alloy SU-31*

Element		W %	Hf %	C %	H ppm	O ppm	N ppm
Ingot Specification #KBI File No. 313-PD2A		16.5-17.5	3.25-3.75	0.09-0.11	15 Max.	225 Max.	100 Max.
Heat No. 1	Electrode Avg.	18.2	3.2	.094	<5	90	134
	Cast Chemistry	---	---	.101	9	50	70

*Program material produced by Kawecki-Berylco Inc. under license from Imperial Metal Industries.

APPENDIX 1B
Chemica Analysis Tabulation
Columb.um Alloy Cb-752**

Element			W %	Zr %	C ppm	H ppm	N ppm	O ppm
Ingot Specification #TWCA-Cb-MS-1			9-11	2-3	150 Max.	15 Max.	100 Max.	225 Max.
Heat No. 1	Electrode	Top	9.7	2.9	100	<5	38	190
		Bottom	9.7	2.5	50	<5	46	100
	Casting		---	---	80	6	63	210

**Program material produced by Teledyne Wah Chang-Albany.

APPENDIX 1C
Chemical Analysis Tabulation
Columbium Alloy C-129Y***

Element			W %	Hf %	Y %	C ppm	H ppm	N ppm	O ppm
Ingot Specification #TWCA-Cb-MS-1			9-11	9-11	.05-.3	150 Max.	15 Max.	100 Max.	225 Max.
Heat No. 1	Electrode	Top	9.6	9.0	.067	60	<5	40	50
		Bottom	9.7	8.5	.074	60	<5	35	50
	Casting		---	---	---	40	13	38	110

***Program material produced by Teledyne Wah Chang-Albany.

APPENDIX 1D
Chemical Analysis Tabulation
Columbium Alloy C-103*

Element			Hf %	Ti %	C PPm	H PPm	N PPm	O PPm
Ingot Specification #TWCA-Cb-MS-1			9-11	.7-1.3	150 Max.	15 Max.	100 Max.	225 Max.
Heat No. 1	Electrode	Top	10.0	.82	45	6	48	170
		Bottom	9.8	.88	40	5	43	150
	Cast Chemistry		---	---	80	10	103	300
Heat No. 2	Electrode Avg.		---	---	80	10	103	300
	Cast		---	.88	70	7	97	330
Heat No. 3	Electrode	Top	9.6	.97	<30	<5	73	180
		Bottom	9.8	.92	<30	<5	68	150
	Cast		---	---	80	13	100	220
Heat No. 4	Electrode	Top	9.3	1.03	30	<5	67	150
		Bottom	9.6	1.01	40	<5	55	190
	Cast		---	---	50	17	82	270
Heat No. 5	Electrode	Top	9.3	1.03	30	<5	67	150
		Bottom	9.6	1.01	40	<5	55	190
	Cast		---	---	<30	23	67	230
Heat No. 6	Electrode	Top	9.3	1.03	30	<5	67	150
		Bottom	9.6	1.01	40	<5	55	190
	Cast		---	---	<30	26	72	210
Heat No. 7	Electrode	Top	9.3	1.03	30	<5	67	150
		Bottom	9.6	1.01	40	<5	55	190
	Cast		---	---	40	9	75	230

*Program material produced by Teledyne Wah Chang-Albany.

APPENDIX 2A

Tensile Properties and NDT
Correlation of C-103 Castings

Sample Number	As-Cast or Heat Treated	Test Temp.	UTS KSI	YS KSI	El. %	R.A. %	ASTM E-155 (Aluminum) Plate No. Corresponding to the Defect Size in Test Bar		
							Gas Hole 1.1 - 1/4"	Shrinkage Cavity 2.1 - 1/4"	Foreign Material 3.12 - 1/4"
7156	As-Cast	R.T.	56.2	44.6	8.0	23.0	1	3	None
7152	As-Cast	R.T.	67.4	47.9	27.5	45.0	None	None	None
7615	As-Cast	R.T.	59.8	45.2	7.5	8.0	2	None	None
7641	As-Cast	R.T.	56.7	44.3	5.5	6.5	None	None	None
7652*	As-Cast	R.T.	60.7	44.3	8.5	7.0	None	None	None
7663	As-Cast Slow Cooled	R.T.	61.3	44.3	14.5	16.0	None	None	None
7664*	As-Cast Slow Cooled	R.T.	62.9	44.1	18.5	24.0	None	None	None
7639	Heat Treated	R.T.	56.9	38.8	19.0	25.0	None	None	1
7636*	Heat Treated	R.T.	60.7	40.1	33.5	53.0	None	None	None
7672	As-Cast + Welded	R.T.	63.4	46.7	10.5	10.0	None	None	None
7673	Welded + Heat Treated	R.T.	59.5	40.3	27.0	54.0	None	None	None
Program Goal		R.T.	40.0	36.0	5.0	--			
7150	As-Cast	2200°F	23.0	14.3	7.0	11.0	None	1	None
7151	As-Cast	2200°F	27.8	22.9	10.5	18.0	None	None	None
7157	As-Cast	2200°F	26.7	23.4	10.0	24.0	None	1	None
7614	As-Cast	2200°F	24.8	17.1	18.0	19.0	None	None	None
7629	Heat Treated	2200°F	23.8	19.6	30.0	30.0	None	None	None
7674	As-Cast + Welded	2200°F	25.1	21.2	5.0	9.0	None	None	None
7675	Welded + Heat Treated	2200°F	23.7	19.6	19.0	26.0	None	None	None
Program Goal		2200°F	20.0	16.5	5.0	--			

APPENDIX 2B

Tensile Properties and NDT Correlation of CB-752 Castings

Rem Sample No.	As-Cast or Heat Treated	Test Temp.	UTS KSI	YS KSI	% El.	R.A. %	ASTM E-155 (Aluminum) Plate No. Corresponding to the Defect Size in the Test Bar		
							Gas Hole 1.1 - ⅛"	Shrinkage Cavity 2.1 - ⅛"	Foreign Material 3.12 - ⅛"
7144	As-Cast	R.T.	67.1	56.4	5.0	5.0	1	1	None
7146	As-Cast	R.T.	67.6	54.8	6.0	11.0	None	1	None
Program Goal		R.T.	40.0	36.0	5.0	---			
7141	As-Cast	2200°F	22.9	19.8	4.5	5.5	None	1	None
Program Goal		2200°F	20.0	16.5	5.0	---			

APPENDIX 2C

Tensile Properties and NDT Correlation of C-129Y Castings

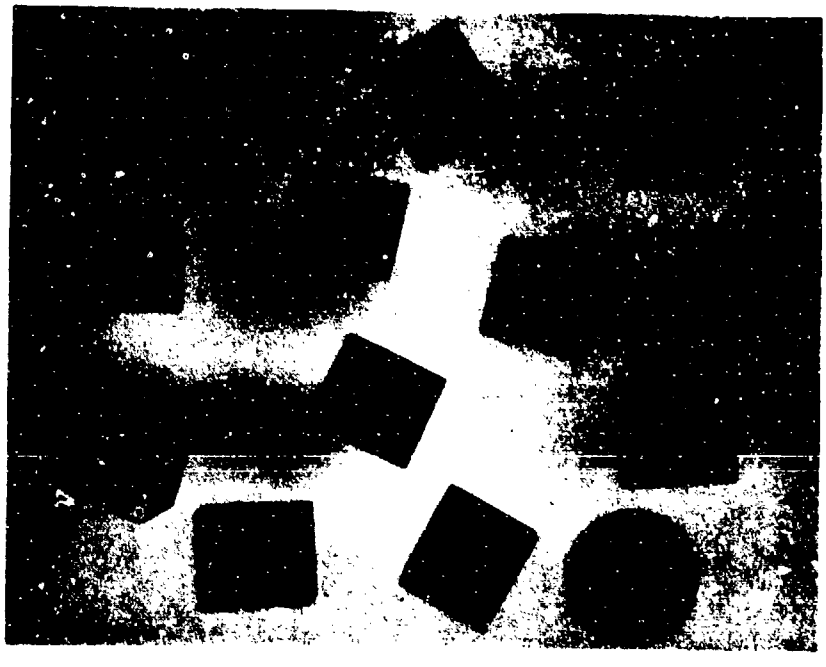
Rem Sample No.	As-Cast or Heat Treated	Test Temp.	UTS KSI	YS KSI	% El.	R.A. %	ASTM E-155 (Aluminum) Plate No. Corresponding to the Defect Size in the Test Bar		
							Gas Hole 1.1 - ⅛"	Shrinkage Cavity 2.1 - ⅛"	Foreign Material 3.12 - ⅛"
7164	As-Cast	R.T.	77.3	67.2	4.5	3.0	1	2	None
7167	As-Cast	R.T.	82.7	65.6	10.5	11.0	None	1	None
Program Goal		R.T.	40.0	36.0	5.0	---			
7171	As-Cast	2200°F	37.3	28.3	8.5	9.0	None	None	None
Program Goal		2200°F	20.0	16.5	5.0	---			

APPENDIX 2D

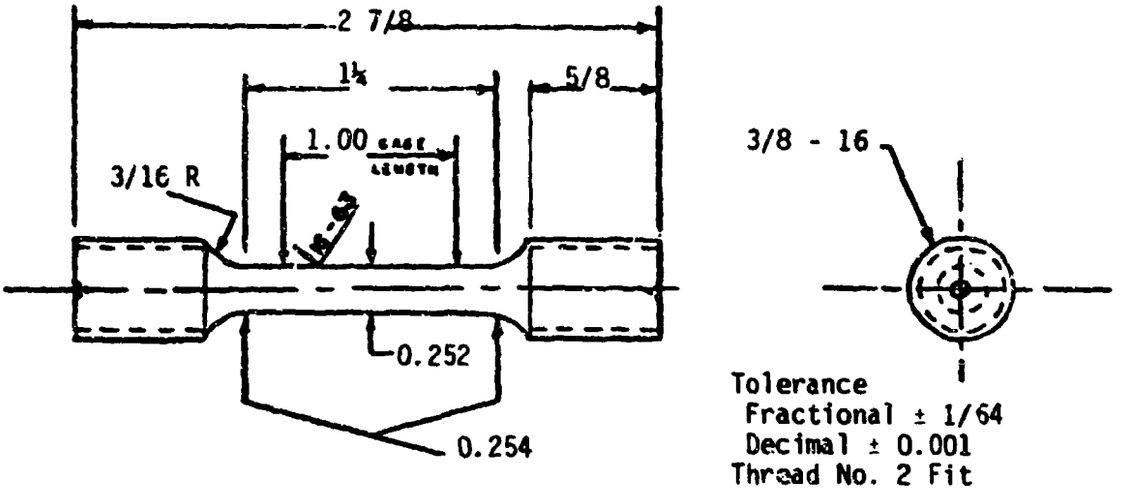
Tensile Properties and NDT
Correlation of SU-31 Castings

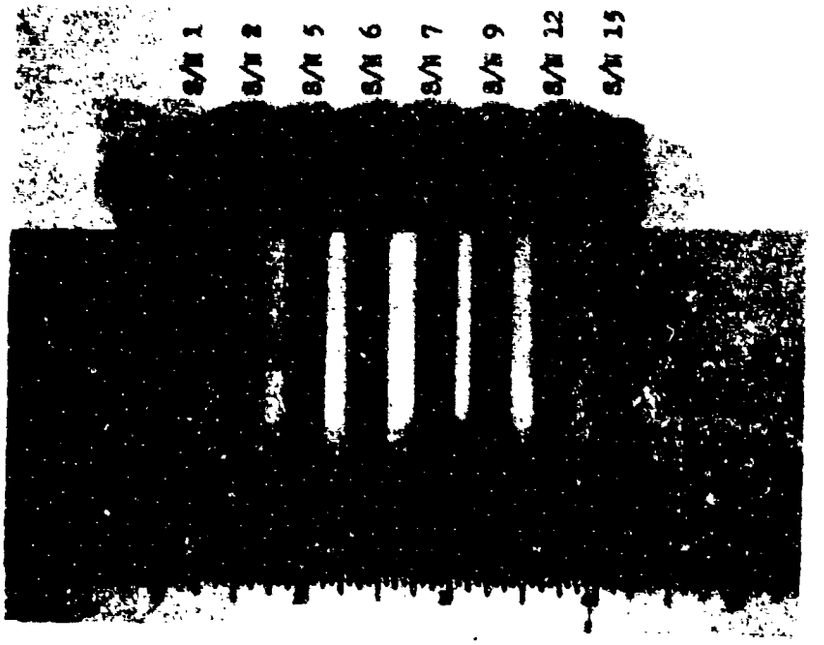
Rem Sample No.	As-Cast or Heat Treated	Test Temp.	UTS KSI	YS KSI	% El.	R.A. %	ASTM E-155 (Aluminum) Plate No. Corresponding to the Defect Size in the Test Bar		
							Gas Hole 1.1 - ¼"	Shrinkage Cavity 2.1 - ¼"	Foreign Material 3.12 - ¼"
7136	As-Cast	R.T.	71.6	---	2.0	1.5	1	1	None
7137	As-Cast	R.T.	79.0	77.4	1.0	1.0	1	2	None
Program Goal		R.T.	40.0	36.0	5.0	---			
7132	As-Cast	2200°F	43.1	35.8	3.5	2.5	None	1	None
7131	As-Cast	2200°F	43.1	22.2	3.0	21.0	1	None	None
Program Goal		2200°F	20.0	16.5	5.0	---			

APPENDIX 3A
Hardness Blanks Columbium Alloy C-103

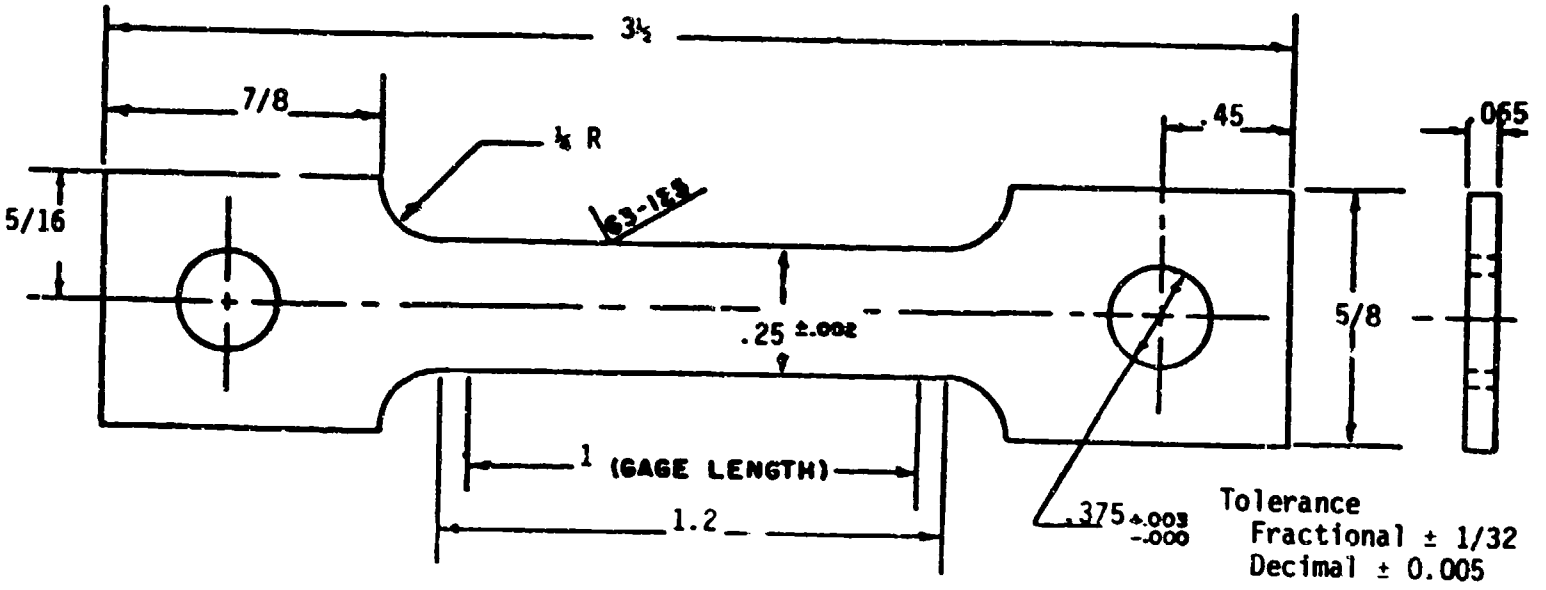


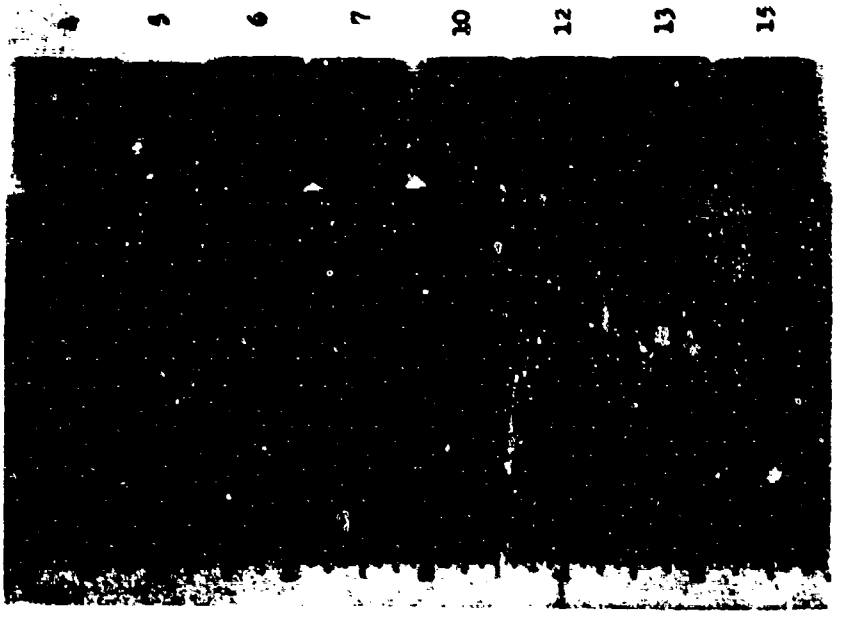
APPENDIX 3B
Round Tensile Specimen Columbium Alloy C-103



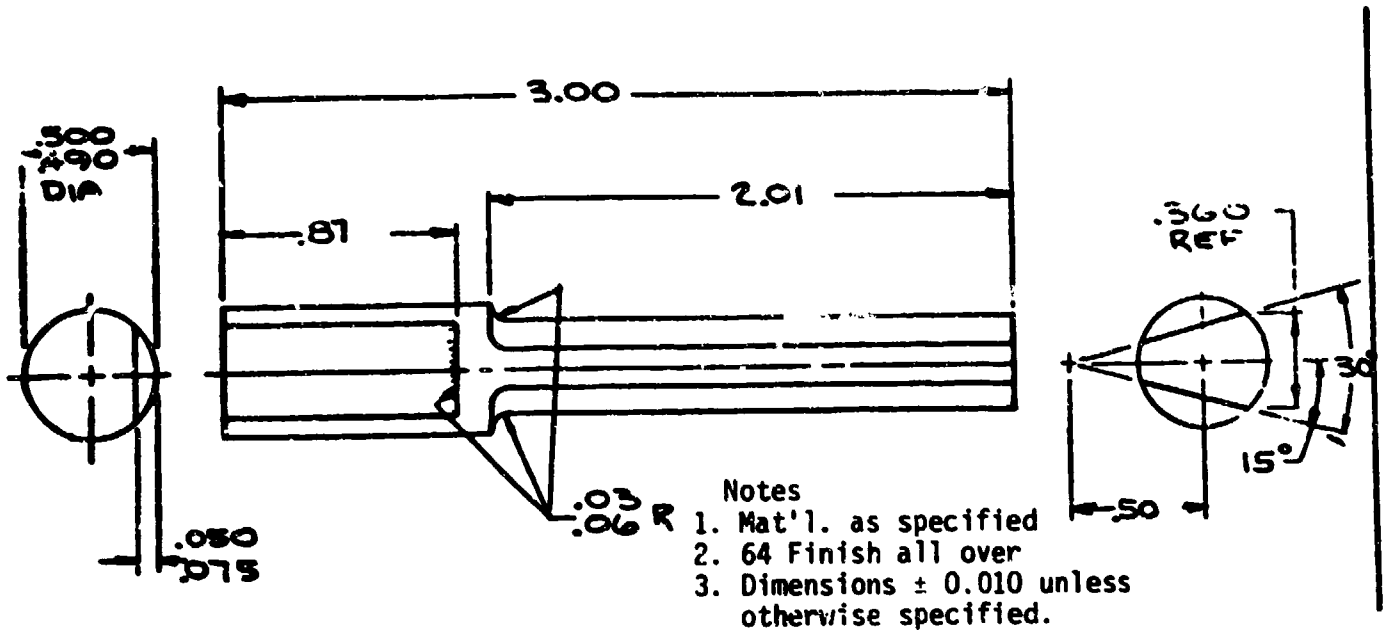


APPENDIX 3C
Flat Tensile Specimen Columbium Alloy C-103

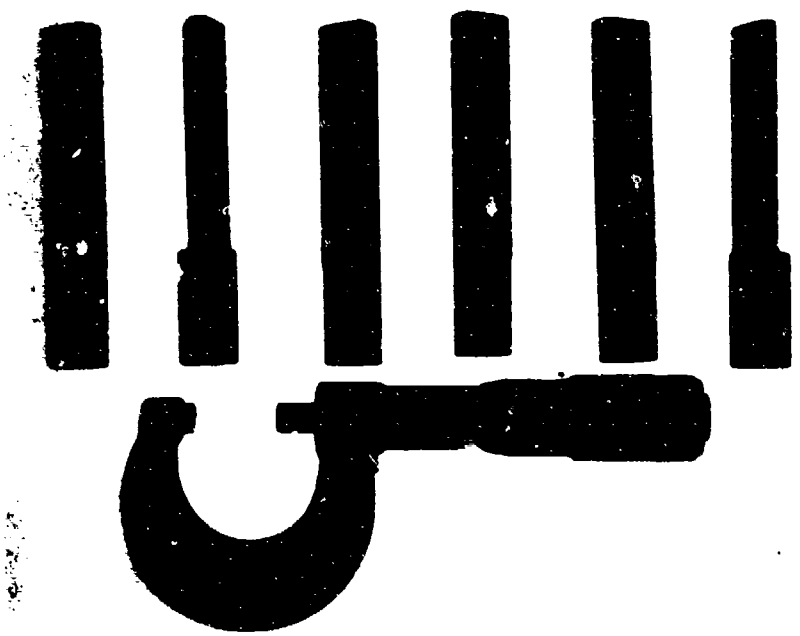




APPENDIX 3D
Oxidation - Erosion Specimen Columbium Alloy C-103



Notes
1. Mat'l. as specified
2. 64 Finish all over
3. Dimensions ± 0.010 unless otherwise specified.



APPENDIX 3E
DRAWING FOR AIRFOIL SPECIMEN

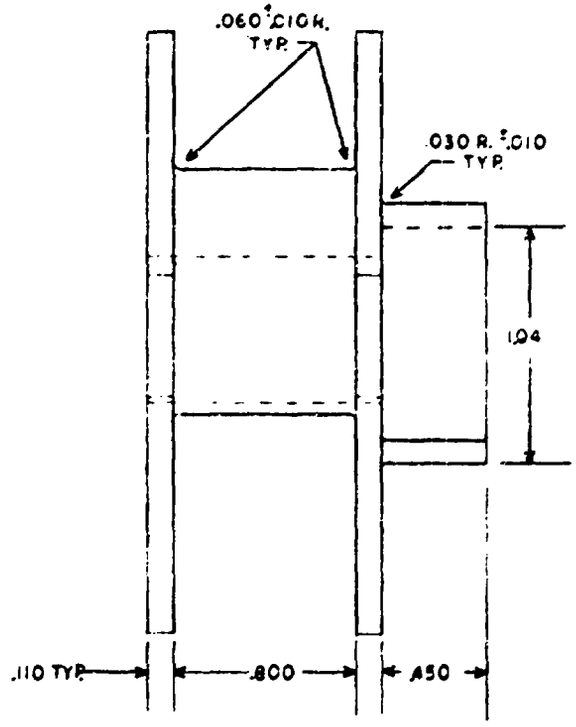
NOTES

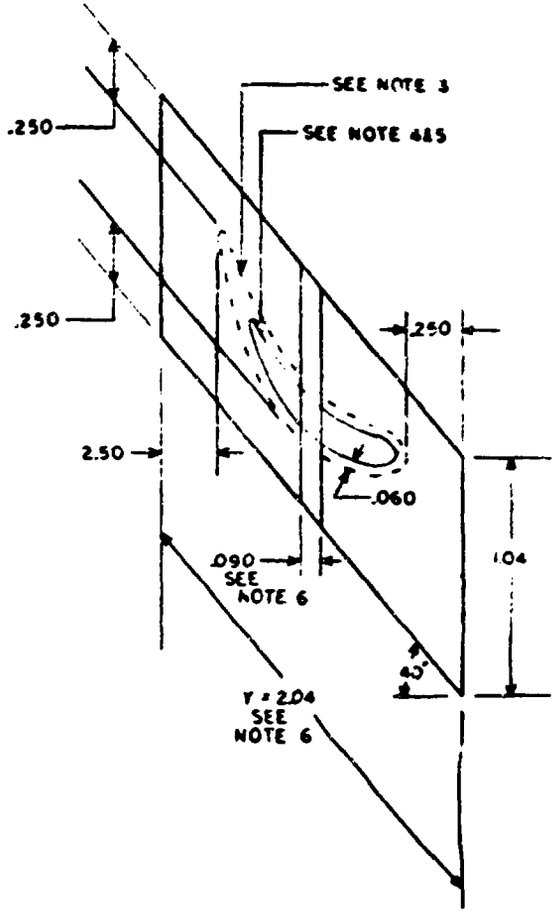
1. Tolerances on dimensions (unless otherwise specified) to be ± 0.010".
2. Radius at corners and edges (unless otherwise specified) to be 1/32".
3. External dimensions of this section to be obtained from drawing of Turbine Vane and glass layout provided.
4. Internal surface of this section to be concentric with external surface except at point nearest to trailing edge. Minimum radius at trailing edge shall be .015/.025.
5. Two pins required in pattern tool to yield .040" and .060" wall.
6. This stub to be positioned at midlength (½ Y).

ALLOWED TOLERANCES

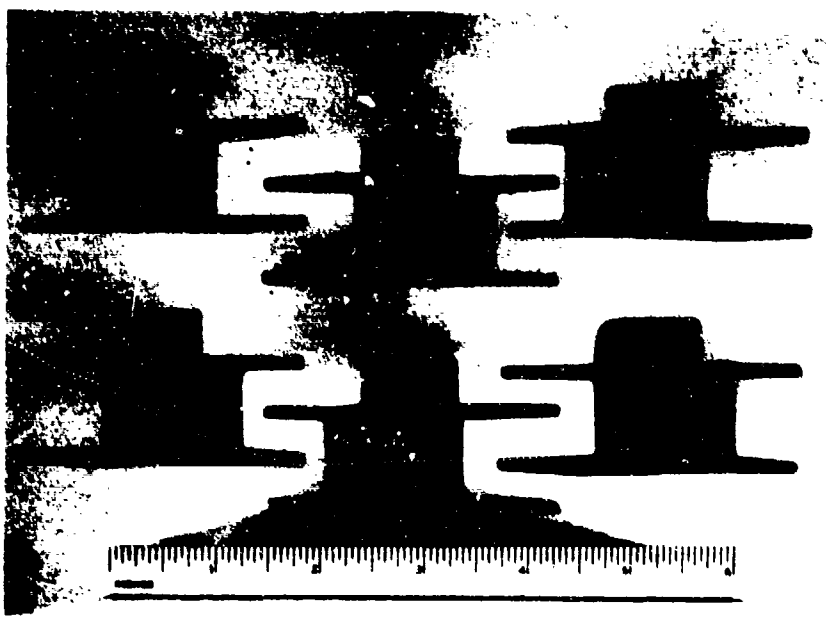
FRACTION (MACH)	±	1/32"
(FAB)	±	1/8"
2 DECIMAL PLACES	±	0.03
3 DECIMAL PLACES	±	0.01
4 DECIMAL PLACES	±	0.0005

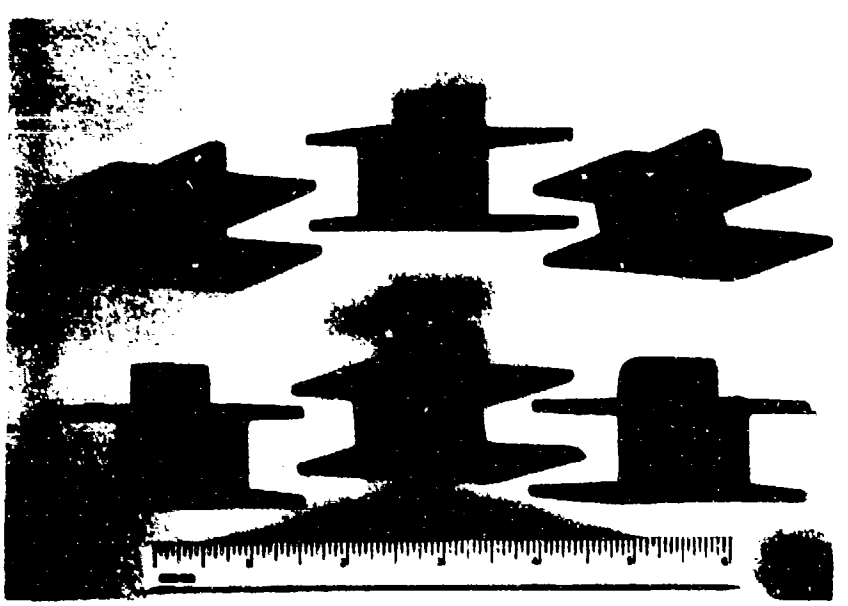
ANGLE: ± 30 MIN.
UNLESS OTHERWISE SPECIFIED





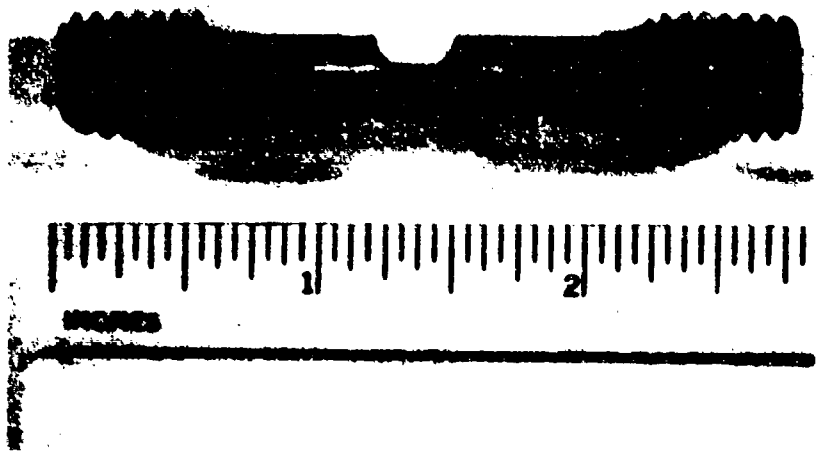
APPENDIX 3F
COLUMBIUM C-103 AIRFOILS





APPENDIX 3G

WELDABILITY TEST SPECIMEN PRIOR TO WELDING



APPENDIX 4

Tentative Specification for Columbium Alloy C-103 Investment Castings

I. Scope and Purpose

This specification incorporates available information relative to the current state of the art and will serve as a basis for formal material and casting specifications which will be required to control procurement of columbium alloy investment castings.

II. Related Documents

The following documents form a part of this specification to the extent they are applicable.

TWCA-Cb-MS-1
Teledyne Wah Chang Albany
Specification for Columbium Alloy

MIL-I-6866
Penetrant Method of Inspection

MIL-C-6021
Castings, Classification and Inspection of

ASTM E8
Standard Method of Tension Testing of Metallic Materials

III. Chemical Composition

Castings shall conform to the following chemical analysis limitations.

Hafnium	9.0 - 11.0%
Titanium	0.70 - 1.3%
Zirconium	.70%-Max.
Tungsten	.0%-Max.
Tantalum	.50%-Max.
Carbon	0.0200% Max.
Oxygen	0.0350% Max.
Nitrogen	0.0150% Max.
Hydrogen	0.0020% Max.

IV. Material Control

A. Virgin Material

Columbium C-103 melt stock shall be prepared by vacuum melting and shall be of composition and uniformity such

that when remelted and poured it will produce castings meeting the chemistry and mechanical property requirements of this specification. Specific processing, chemical composition, and physical form of virgin material shall be as agreed upon by the foundry and the material supplier.

B. Revert Material

Revert material consists of gates, risers, sprues, crucible skulls, and rejected castings which, when remelted with or without addition of virgin material, will produce castings meeting the chemical and mechanical property requirements of this specification.

C. Traceability

The casting vendor shall maintain a system suitable to control and document the material history of each casting produced and to assure that the required chemical and mechanical properties are met. Interstitial chemical analysis and tensile properties shall be determined for each pour unless adequate process repeatability data has been accumulated and a lesser frequency is agreed upon between purchaser and supplier.

V. Mechanical Properties

A. Specimens machined from cast tensile specimen blanks, from associated gating, or cut from actual components shall exhibit the following minimum tensile properties at room temperature:

	Cast to Size Tensile Specimens	Machined Gating or Castings
Ultimate Tensile Strength	54,000 PSI	54,000
Yield Strength (0.2% Offset)	38,000 PSI	38,000
Elongation (in 4D)	20%	10%

B. Heat treatment and/or "in situ" heat treatment during a coating cycle may be applied to produce a finished component or assembly which meets the minimum mechanical property requirements.

VI. Hardness

A. The maximum hardness shall be Rockwell B 95.

B. Hardness of the finished casting shall not vary from edge to center or in various locations by more than 25 Rockwell B numbers or equivalent.

VII. Non-Destructive Testing

A. Dimensional inspection shall be performed to assure conformance to drawing requirements. A complete dimensional analysis shall be performed on initial castings produced following a change in tooling, gating, or other process factors which may affect dimensions. Production castings shall be checked 100% for selected control dimensions which would indicate variation in casting size.

B. Penetrant Inspection

Finished castings shall be 100% inspected for deep surface discontinuities using the fluorescent penetrant technique according to MIL-I-6866. Cracks, laps, seams, or other linear or linearly oriented defects will not be acceptable. Pinholes or other defects greater than a diameter agreed upon by purchaser and vendor will not be acceptable.

C. Radiographic Inspection

Castings shall be 100% radiographically inspected according to MIL-C-6021 and shall meet the grade specified on the engineering drawing for the area being inspected.

D. Visual Inspection

Castings shall be inspected visually to verify that castings are clean, free of foreign material, and exhibit good workmanship.

VIII. Repair of Defects

A. Surface discontinuities may be removed by grinding provided drawing tolerance limits are not exceeded. When dimensional limits are violated, or for all rejectable internal discontinuities, repair shall be done by TIG welding using filler metal meeting the same chemical composition limits as for C-103 electrode material.

B. Welding shall be performed using a process which has been demonstrated capable of preparing weld test specimens which meet the requirements for tensile properties shown in paragraph IV for master heat qualification for specimens machined from castings. The weld test specimen shall consist of a 0.252 inch round tensile bar from which 50% of the cross section area has been removed from one side of the center of the gage section and which has been welded and optionally post-weld heat treated prior to final machining and testing.

IX. Certification

Certifications covering applicable chemical analysis, mechanical property, and NDT inspection results shall be submitted with each shipment of castings.